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**BARTELLS**

**An Attempt to Reduce the Drying  
Shrinkage & Cracking of two Washington  
Clays; with some Investigations  
on their Refractory Properties**

**Ceramics**

**B. S.**

**1913**

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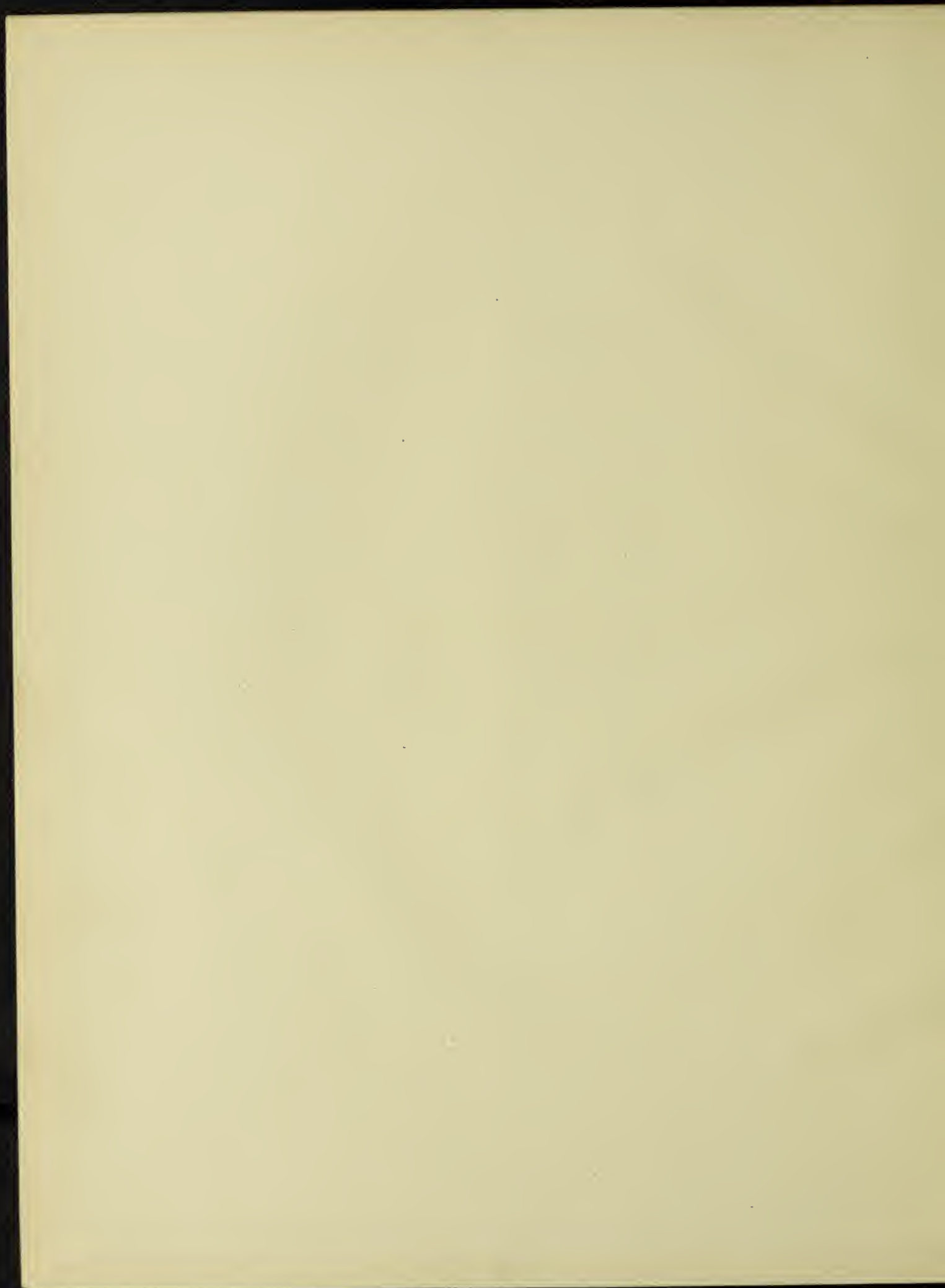


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**AN ATTEMPT TO REDUCE THE DRYING SHRINK-  
AGE AND CRACKING OF TWO WASHINGTON  
CLAYS; WITH SOME INVESTIGATIONS ON  
THEIR REFRACTORY PROPERTIES**

BY

**HENRY HARRISON BARTELLS**

---

**THESIS**

FOR THE

**DEGREE OF BACHELOR OF SCIENCE**

IN

**CERAMICS**

---

**COLLEGE OF SCIENCE**

**UNIVERSITY OF ILLINOIS**

**1913**



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June 3d 1913

THIS IS TO CERTIFY THAT THE THESIS PREPARED UNDER MY SUPERVISION BY

Henry Harrison Bartels

ENTITLED *An Attempt to Reduce the Drying Shrinkage and Cracking of two Washington Clays; With Some Investigations on their Refractory Properties.*

IS APPROVED BY ME AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE

DEGREE OF *Bachelor of Science* in  
*Ceramics*

*A V Bleiringer*  
Instructor in Charge

APPROVED: *R T Stull*

HEAD OF DEPARTMENT OF *Ceramics*

247342

Small camp

March 10, 1884

Left camp at 8 AM. and went to the  
spring. The water is very good and  
the spring is very large. The water is  
very good and the spring is very large.

March 11, 1884

Left camp

March 12, 1884

Left camp

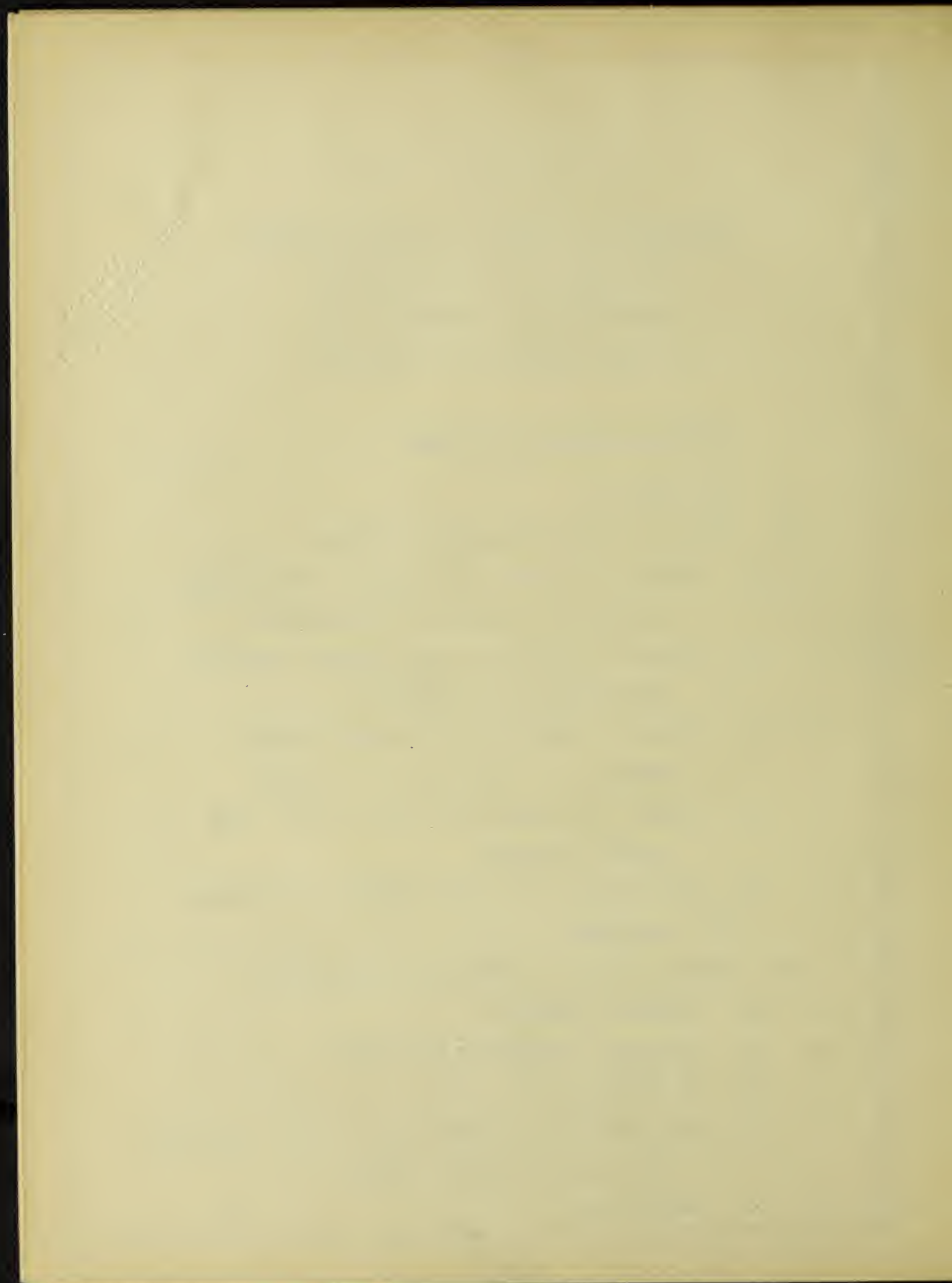
March 13, 1884

AN ATTEMPT TO REDUCE THE DRYING SHRINKAGE  
AND CRACKING OF TWO WASHINGTON  
CLAYS; WITH SOME INVESTIGATIONS  
OF THEIR REFRACTORY PROPERTIES.

Outline of Method of Attack.

1. Introduction.
2. Study of Drying Shrinkage by Volume.
  - A. Effect of Addition of Reagents upon Drying, Cracking and Reduction of Shrinkage.
  - B. Effect of Preheating upon Drying, Cracking and Reduction of Shrinkage.
  - C. Effect of Addition of Grog upon Drying, Cracking and Reduction of Shrinkage.
  - D. Effect of variation of Water content upon volume Shrinkage.
  - E. Discussion of the three methods of Reducing Shrinkage.
3. Investigations of Refractory Properties.
  - A. Chemical Analysis.
  - B. Softening Point Determinations.
  - C. Load Test.
  - D. Determination of Vitrefication Range, including Porosity-Temperature Curves.
  - C. Conclusions.





## INTRODUCTION.

The names of these two clays are Taylor Fire Clay and New #3 Buff. They are both mined underground and are so closely related that it is sometimes difficult to distinguish any difference in their appearances. The two samples considered in this work, however, are quite different in appearance although their chemical analysis and physical properties are very similar. The Taylor Clay is a buff colored clay, very plastic and shows a high shrinkage. The #3 Buff Clay has a darker color caused by a high content of carbonaceous matter. It contains streaks of limonite, is very plastic and has a high shrinkage. Both clays have a decided tendency to warp, check and crack in drying. The drying must be done very slowly and even after the utmost care is taken, cracking is noticed in many cases, so that the dried trials are cracked and checked throughout the whole mass and can be easily shattered between the fingers. This checking is increased in the burning and it was the writer's experience that when 100% raw clay was used, trials could not be dried and burned without checking. Quite a little difficulty was encountered in oxidizing the #3 Buff Clay. However outside of the content of carbonaceous matter which accounts for the difference in color, the clays are quite similar in all respects and for this reason some of the tests will be applied only to the Taylor Clay.

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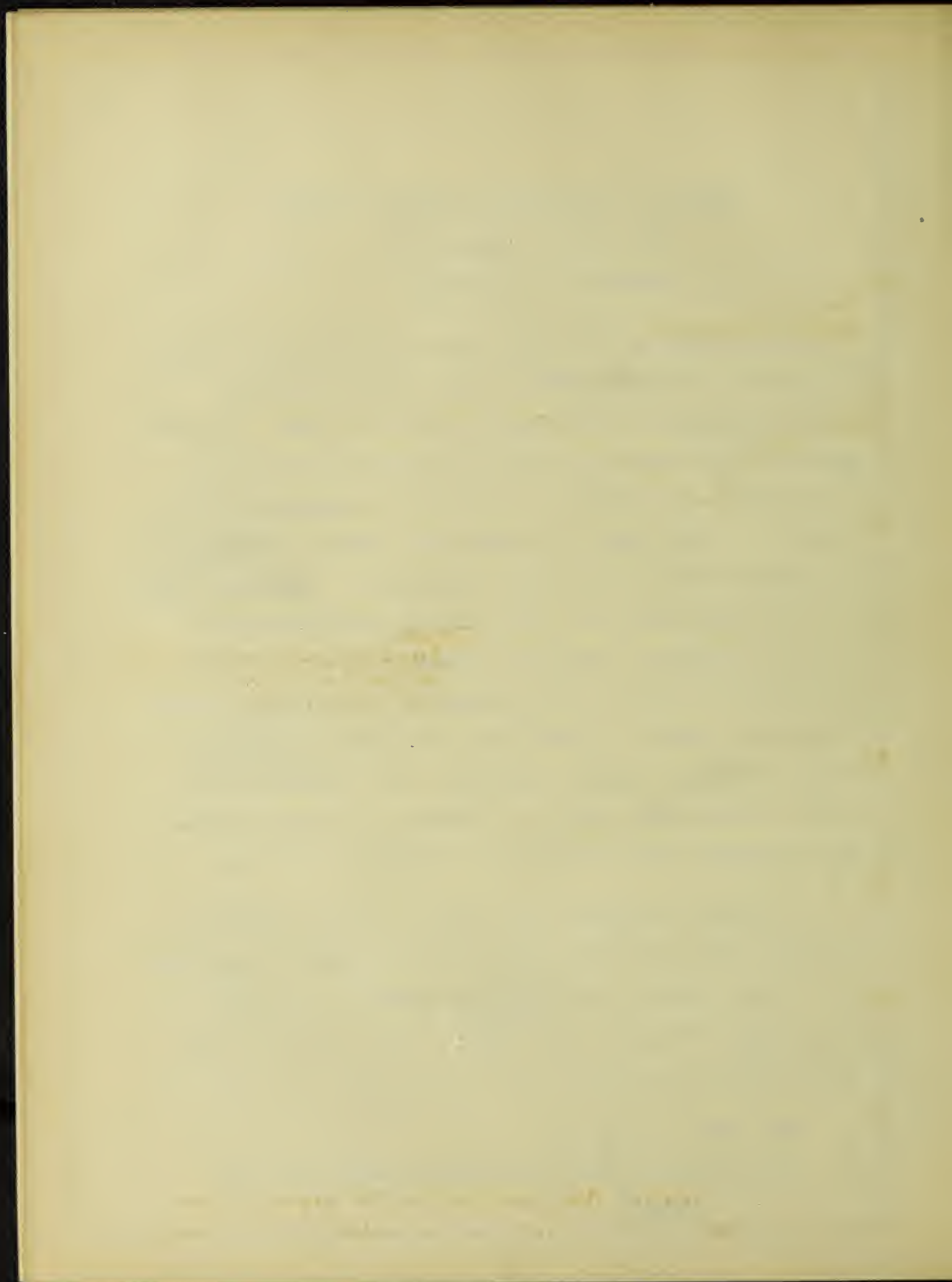


EFFECT OF ADDITION OF CHEMICAL REAGENTS  
UPON DRYING, CRACKING AND  
REDUCTION OF SHRINKAGE.

Investigations along these lines have been carried on by Rohland<sup>1</sup> and Bleininger<sup>2</sup>, but the work has not been extensive enough to establish any clear cut laws. Rohland arrives at the conclusion that the plasticity of clay is increased by the presence of "H" ions and decreased by the presence of "OH" ions. In many cases however, Bleininger's results contradicted Rohland's statements. Bleininger found that the conditions were quite complex in the clays and that the increase or decrease in plasticity was no doubt influenced very much by the presence of the different salts contained in them. He found that the electrolytes NaCl, CaCl<sub>2</sub>, and AlCl<sub>3</sub> in small quantities had a tendency to increase the shrinkage but as the amounts of the electrolyte were increased they decreased the shrinkage materially.

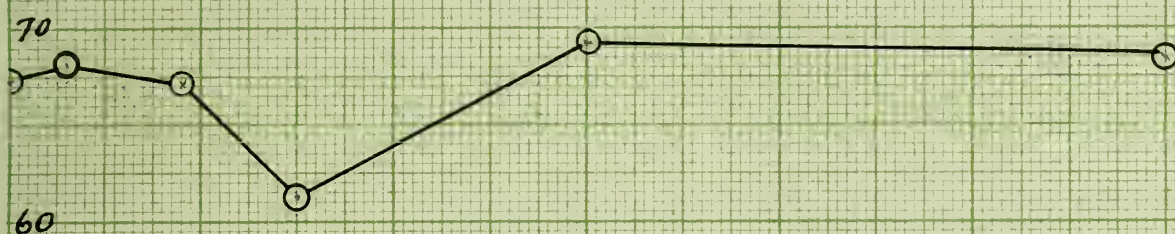
In order to determine the effect of these reagents on the Taylor Clay, they were all made up into Normal solutions, the required amount of solution was added to the sample and then enough additional water added to make the total water

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1. "Die Tone," pp 35-19.
  2. "Transactions American Ceramic Society, " Vol. XIV.  
pp. 827-839





Percent  
Volume Shrinkage.



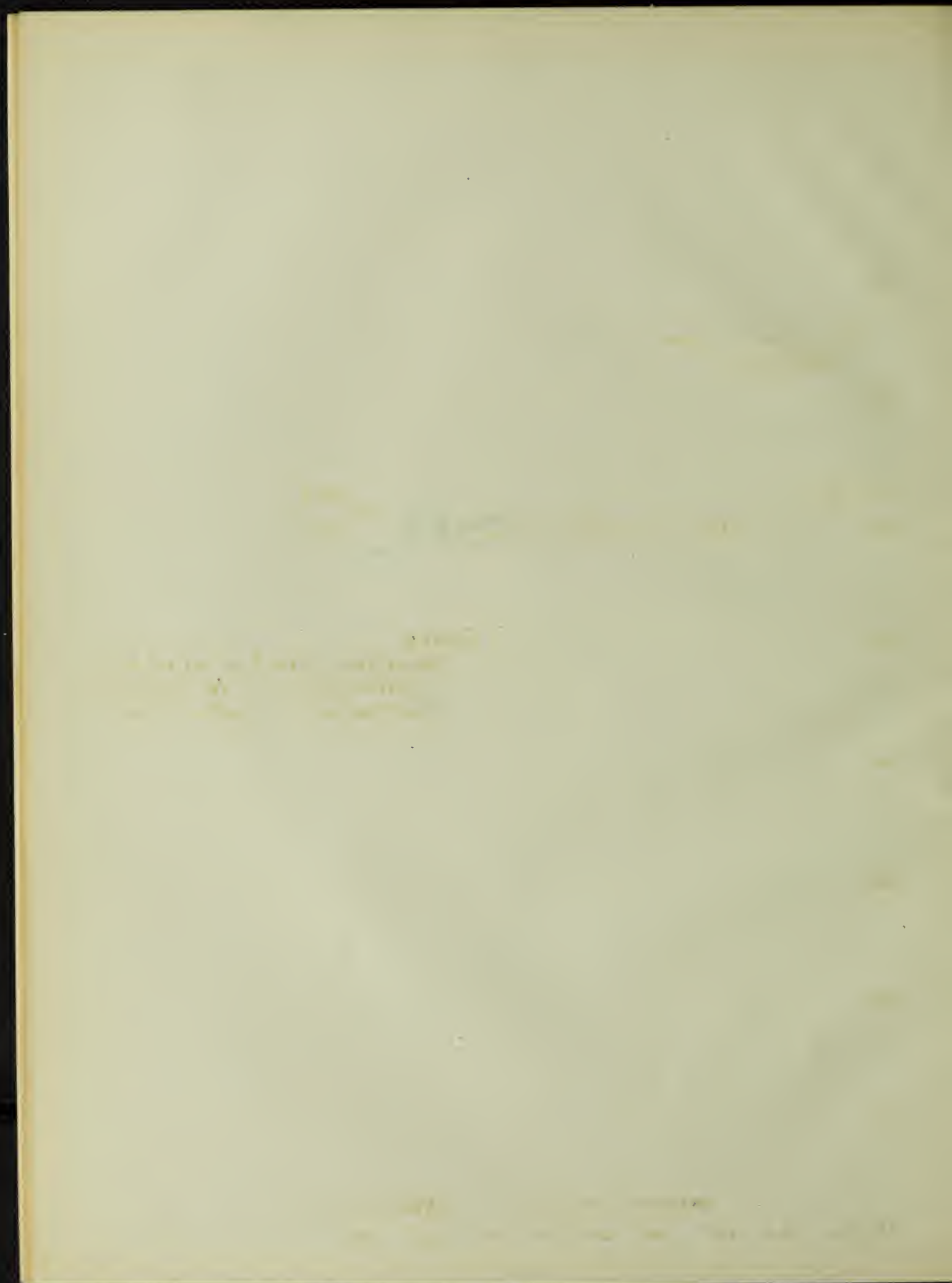
Curve

Showing effect of addition  
of NaCl upon the Shrinkage  
of Taylor Clay

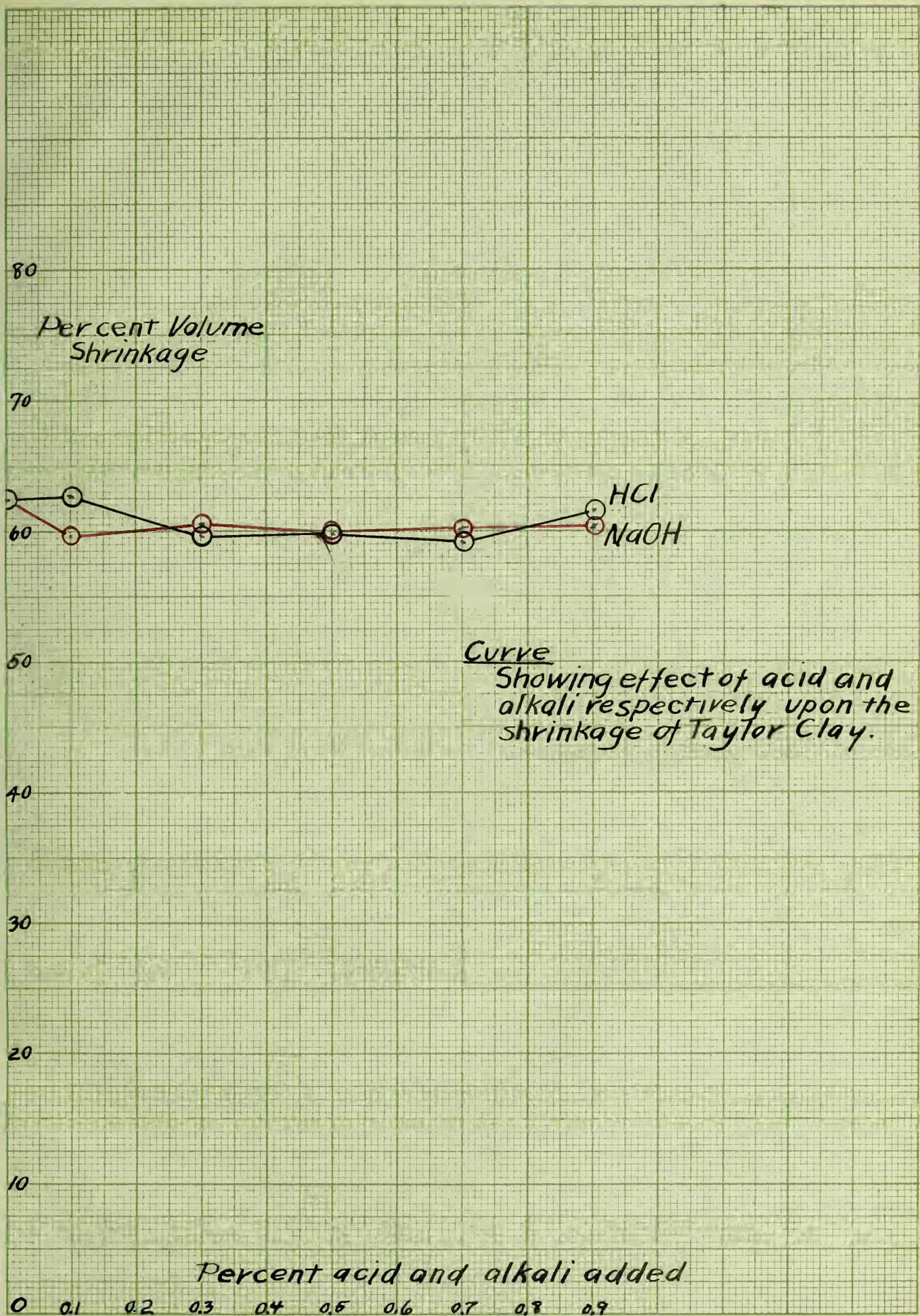
Grams NaCl added to 100 Grams Clay.

0 0.5 1.0 1.5 2.0

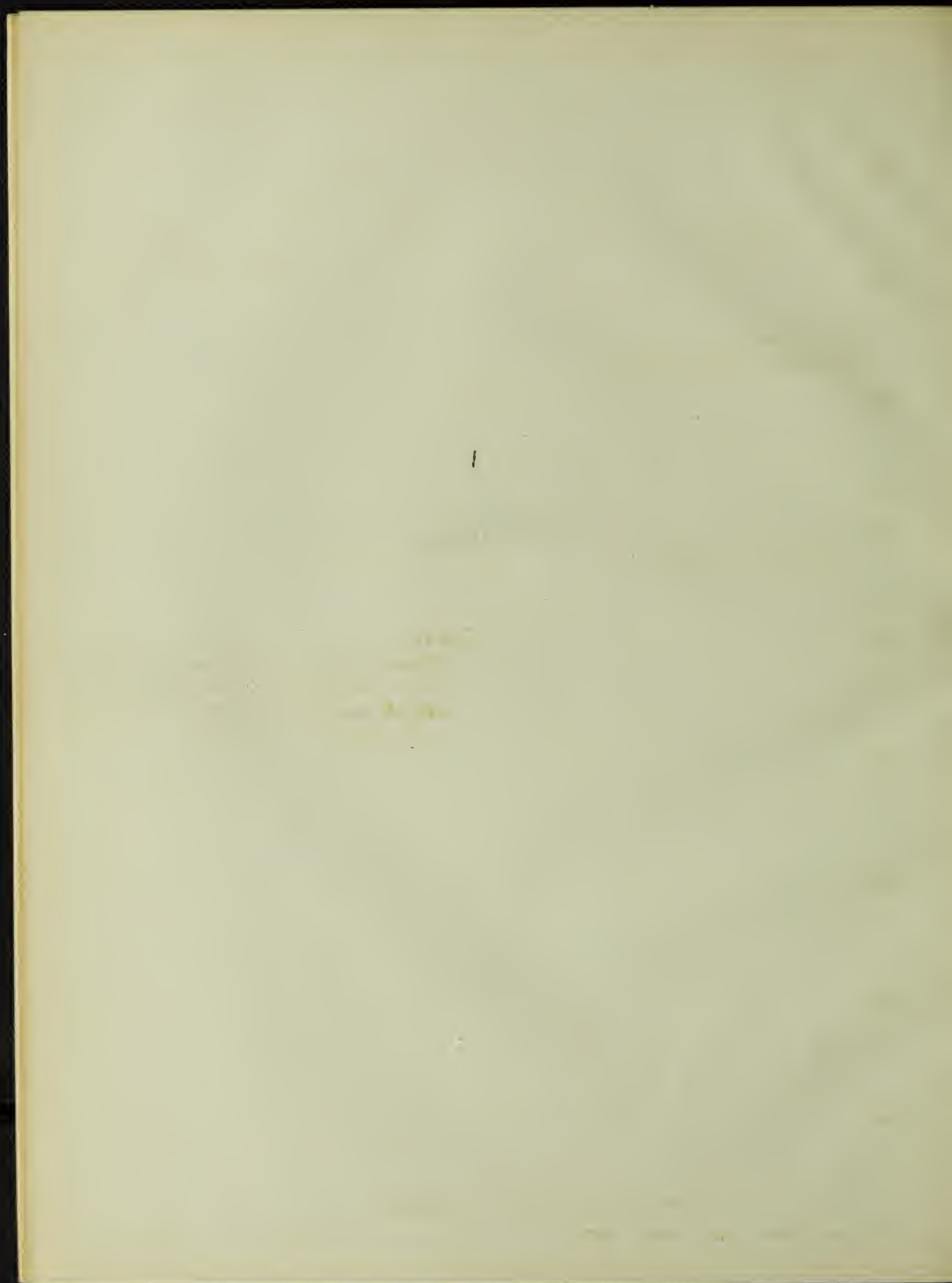




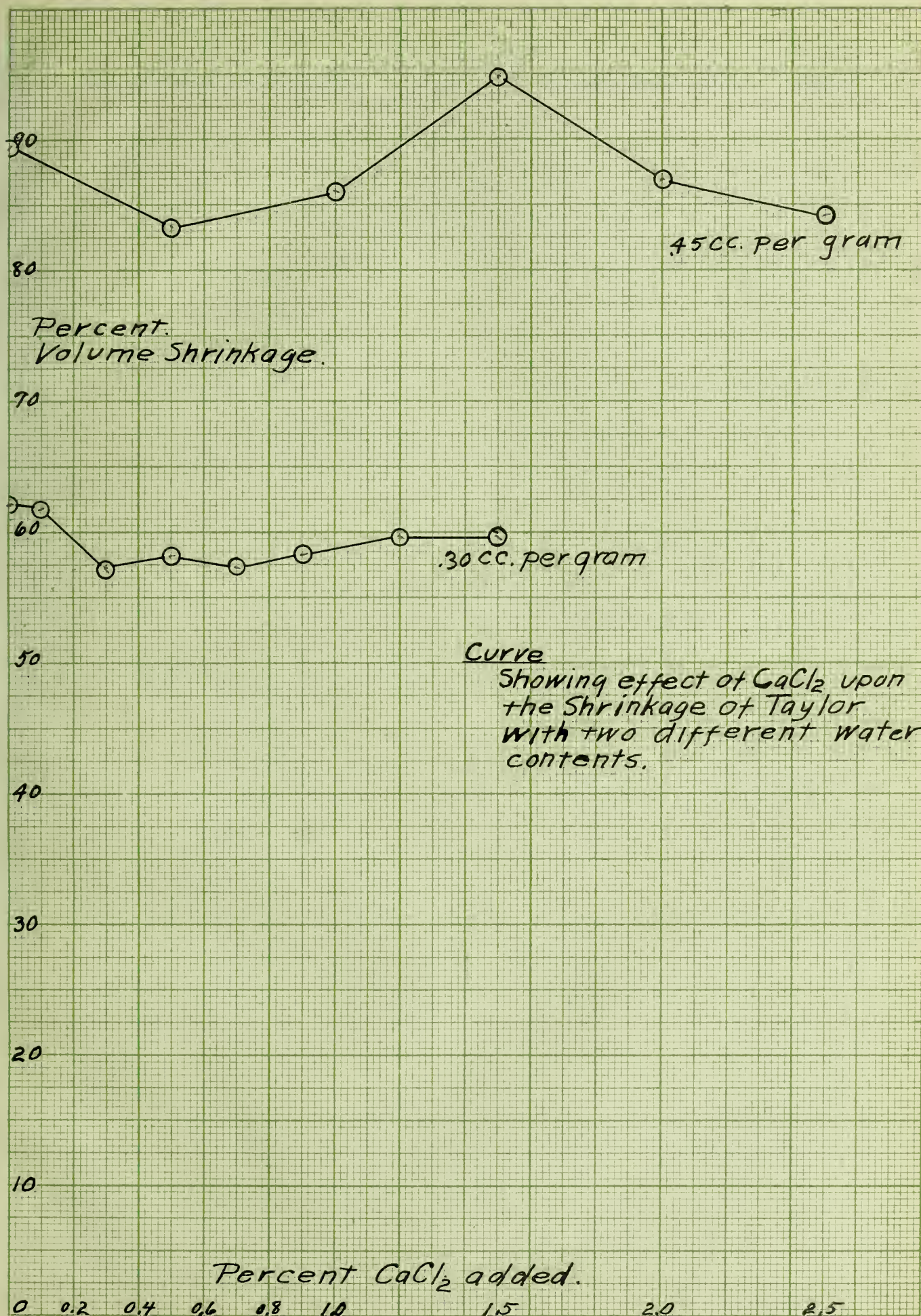


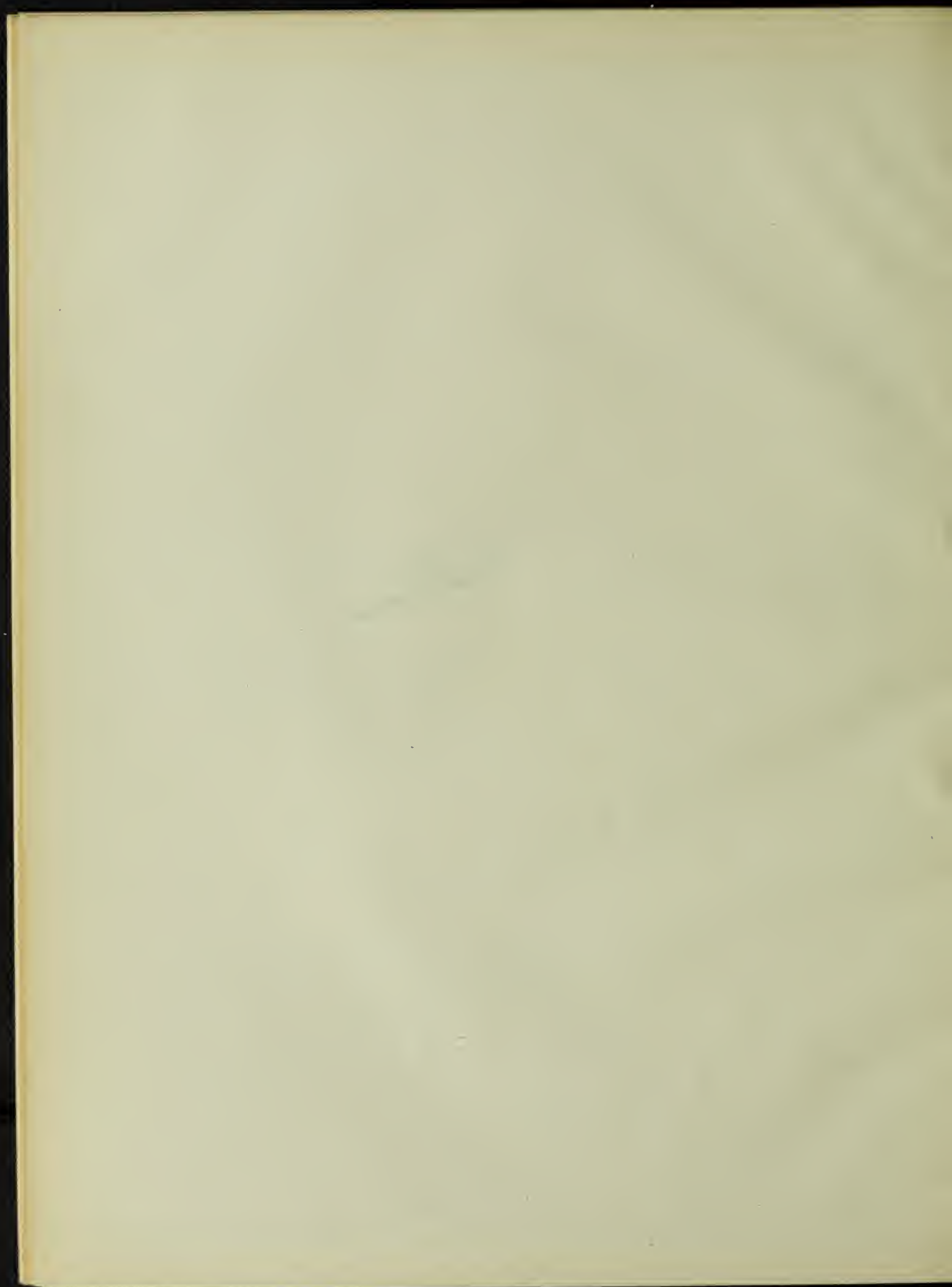












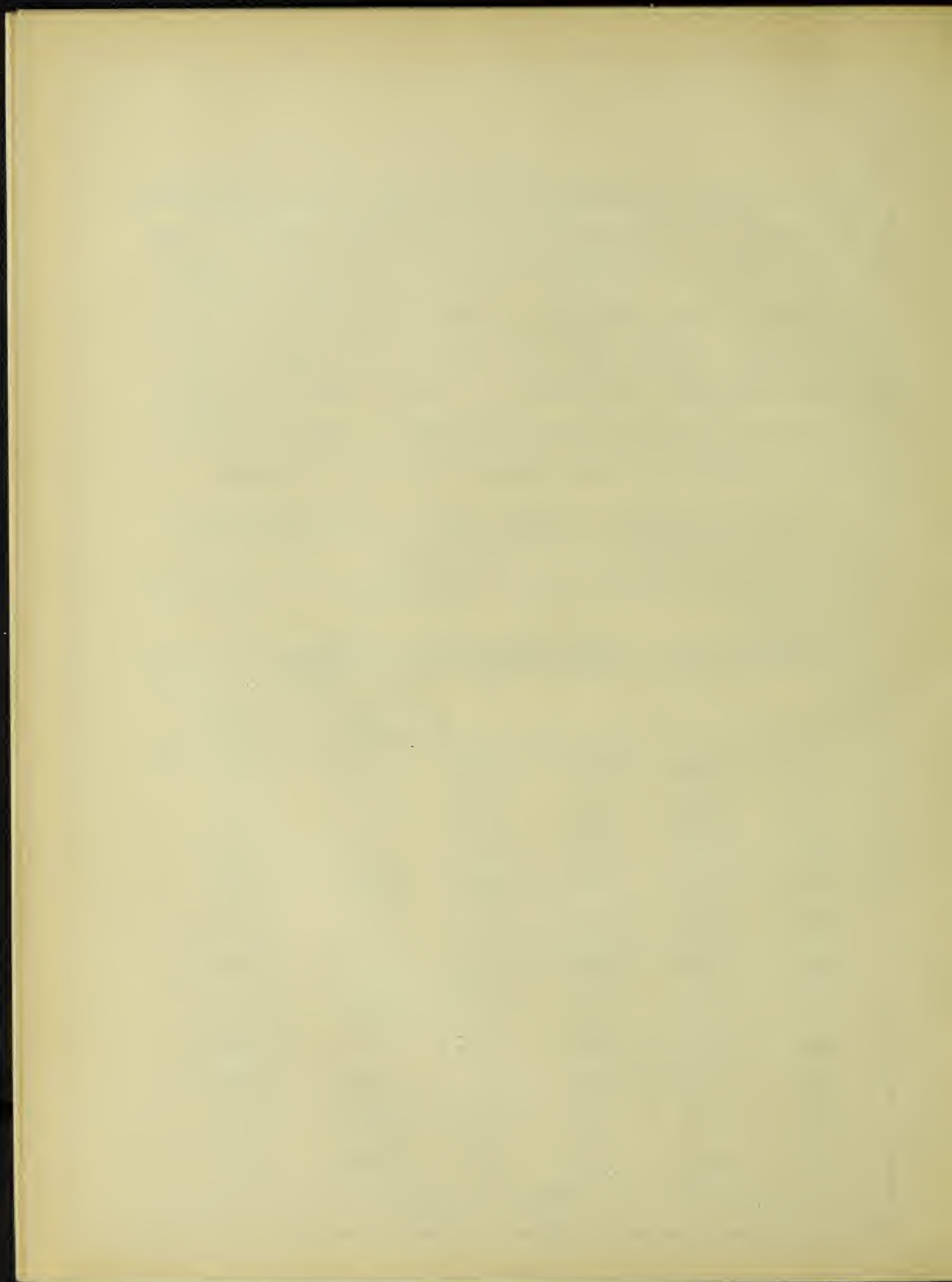


content 18 cc per 60 grams of clay, which had previously been determined as the proper proportion for good working. One series of work with  $\text{CaCl}_2$  using 27 cc per 60 grams of clay was made up and in this way a higher per cent. of salt was introduced. After adding the reagents the samples were wedged thoroughly and placed in a moisture can for twenty-four hours to improve their uniformity. The results are shown graphically by the accompanying curves and data.

Table of effects of acid, alkali, and electrolyte on the volume shrinkage of Taylor Fire Clay:

Per cent. of Reagent.	Per cent. Volume Shrinkage.				
	HCl	NaOH	NaCl	$\text{CaCl}_2$	
				.45 cc per gram	.30 cc per gram
0.0	62.23	62.23	67.34	89.67	62.23
0.1	62.59	59.61	68.11	-----	61.83
0.3	59.71	60.59	66.77	-----	57.05
0.5	59.81	59.90	62.01	83.33	58.29
0.7	59.03	60.12	-----	-----	57.39
0.9	61.65	60.27	-----	-----	58.30
1.0	-----	-----	69.36	86.01	-----
1.2	-----	-----	-----	-----	59.63
1.5	-----	-----	-----	94.90	59.47
2.0	-----	-----	68.38	86.91	-----
2.5	-----	-----	-----	84.18	-----





It will be noticed that in the cases of the NaCl, NaOH and  $\text{CaCl}_2$  where the same total water content was employed; the curves follow the same general lines. There is first a decrease in shrinkage, after which it increases, after which it is uniform. With HCl the first decrease in shrinkage appears but there is no increase afterwards as in the case of the other two. Using  $\text{CaCl}_2$  with a high water content a well defined max point is noticed at 1.5%

In all cases the changes are only slight and show that the addition of reagents will have no practical value in reducing the shrinkage of the clay.

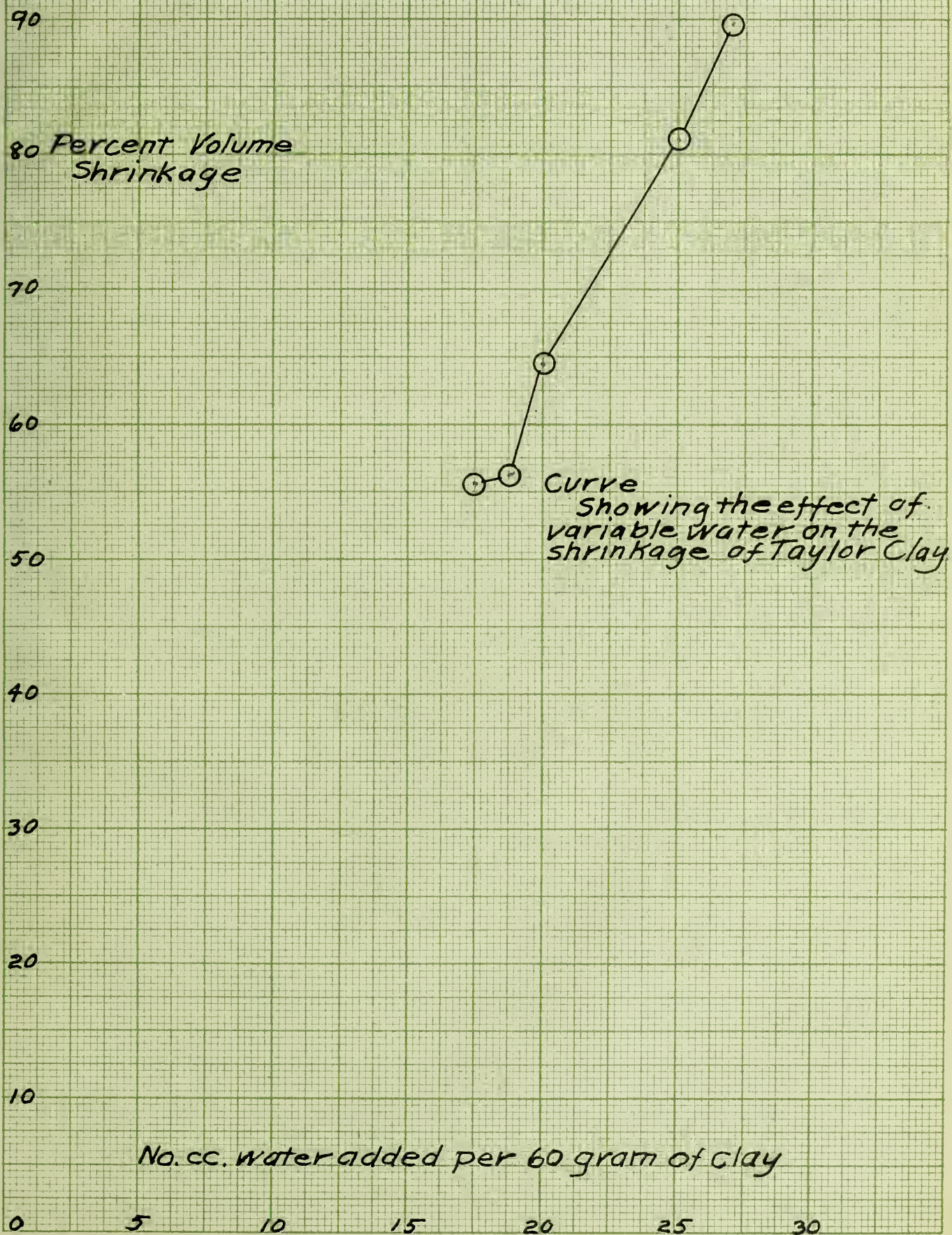
#### EFFECT OF VARIATION OF WATER CONTENT ON VOLUME SHRINKAGE.

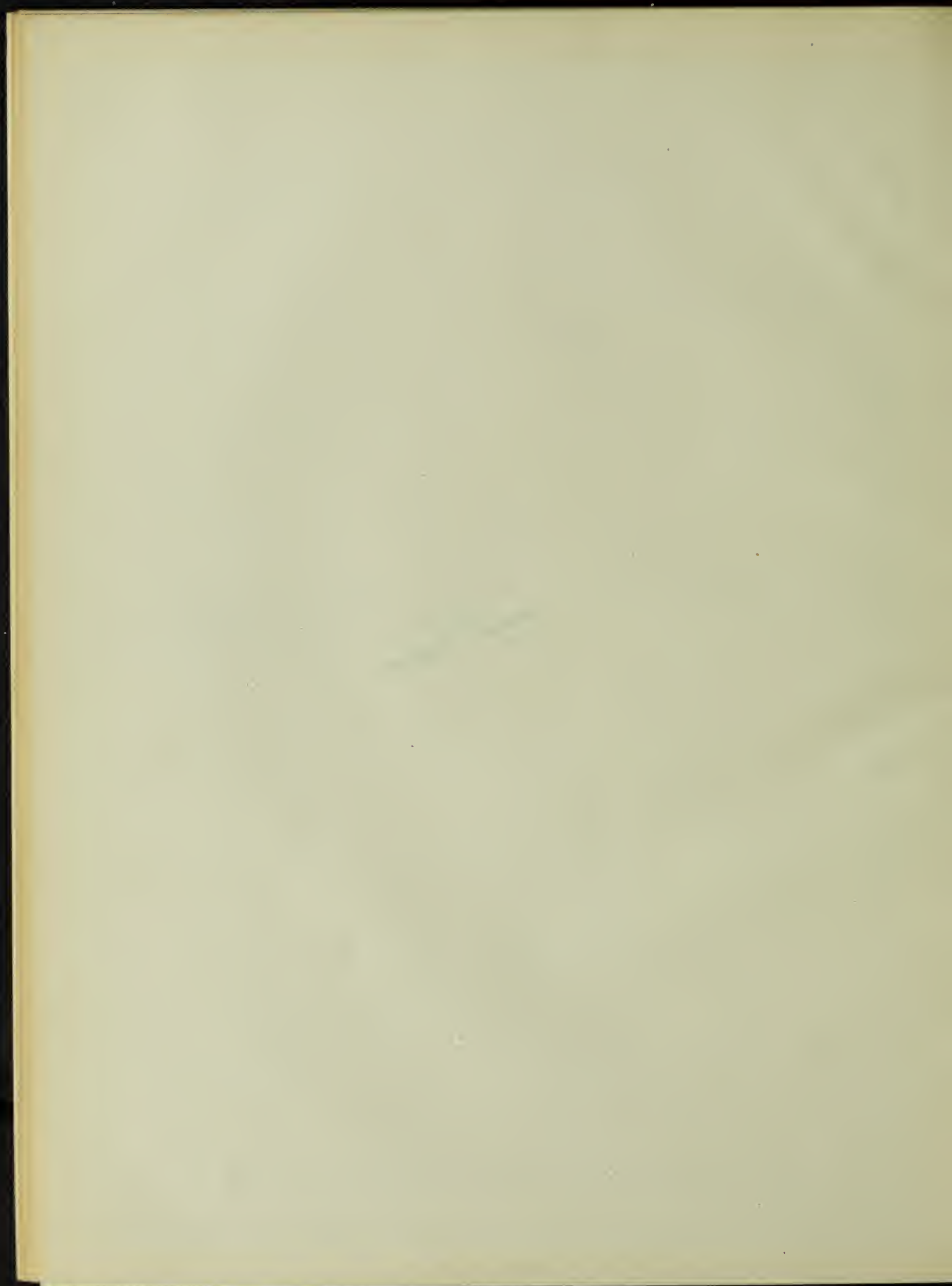
It was noted that variation of the amount of water added to a clay had a marked effect upon its volume shrinkage. In order to study this effect, trial pieces were made using different amounts of water. The clay sample used was 60 grams and the water was varied from as small an amount as could be used (17.5 cc. up to a content of 27 cc. where the brickette was very sticky and soft). The

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greatest difference in shrinkage was 33.54%, the shrinkage increasing as the water increased. The data and curves will show the result of this test.

Table of effect of variation of water content  
on shrinkage:

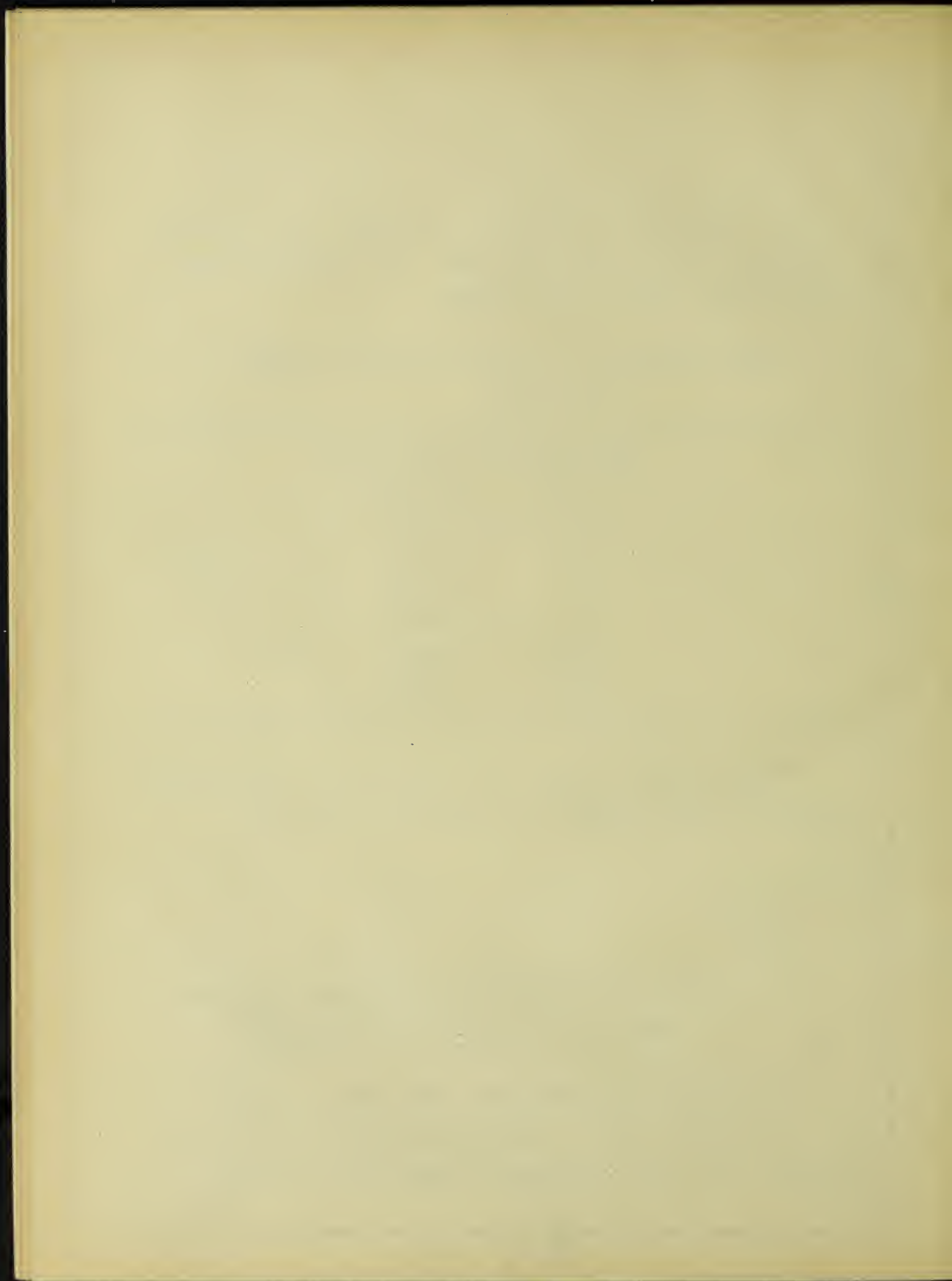
Water added in ccm.	Per cent volume Shrinkage.
17.5	55.81
18.7	55.94
20.0	64.55
25.0	81.19
27.0	89.67

These results emphasize very forcibly the necessity of keeping the water content at a minimum at all times.

#### A STUDY OF PREHEATING WITH REFERENCE TO DRYING, CRACKING, AND REDUCTION OF SHRINKAGE.

It is a well known fact that when clays are heated they lose plasticity, the amount lost depending on the intensity of the heat and length of time they are heated;

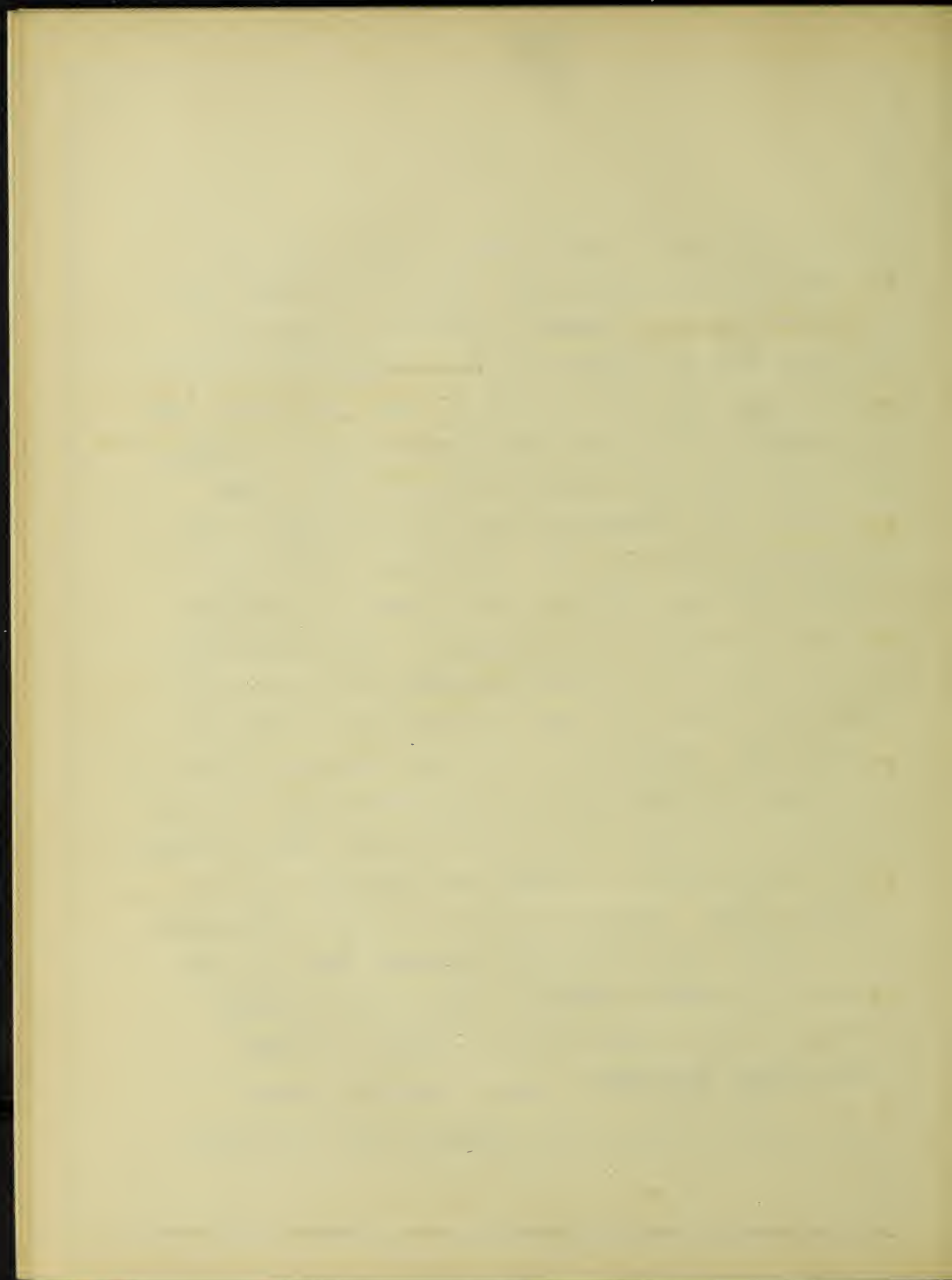




and if this heat is carried above a certain point they lose all of their plasticity. Working on the hypothesis that shrinkage is a measure of plasticity, samples were preheated at 150 degrees C and above at intervals of about 50 degrees, until they had lost all their plasticity. All preheating at the higher temperatures was done in a small, circular, gas-fired, laboratory kiln, and temperatures measured with a Thermo-couple and milli-voltmeter. The preheating done at the lower temperatures was done in a small, sheet iron, laboratory oven, heated by a Bunsen gas burner and regulated by a Thermostat. These temperatures were raised to the required temperature and held there for two hours, after which they were placed in an airtight jar until used. Brickettes were made from them using 18 cc of water per 60 grams of clay, the brickettes were weighed, immersed in kerosene for six hours and their volumes measured in a small laboratory volumeter using kerosene as the immersing fluid. They were dried in air, then at 120 degrees C., weighed and again immersed in kerosene for six hours and the dry volumes measured. The per cent. shrinkage was determined in terms of the dry volume as follows:

$$\frac{\text{Wet Volume} - \text{Dry volume}}{\text{Dry Volume}} \times 100 = \% \text{ Drying Shrinkage.}$$

The results are given in the following tables and curves:





Percent  
Volume Shrinkage

Curves

Showing effect of Preheating  
upon the Volume Shrinkage  
of New #3 Buff and Taylor  
Clays.

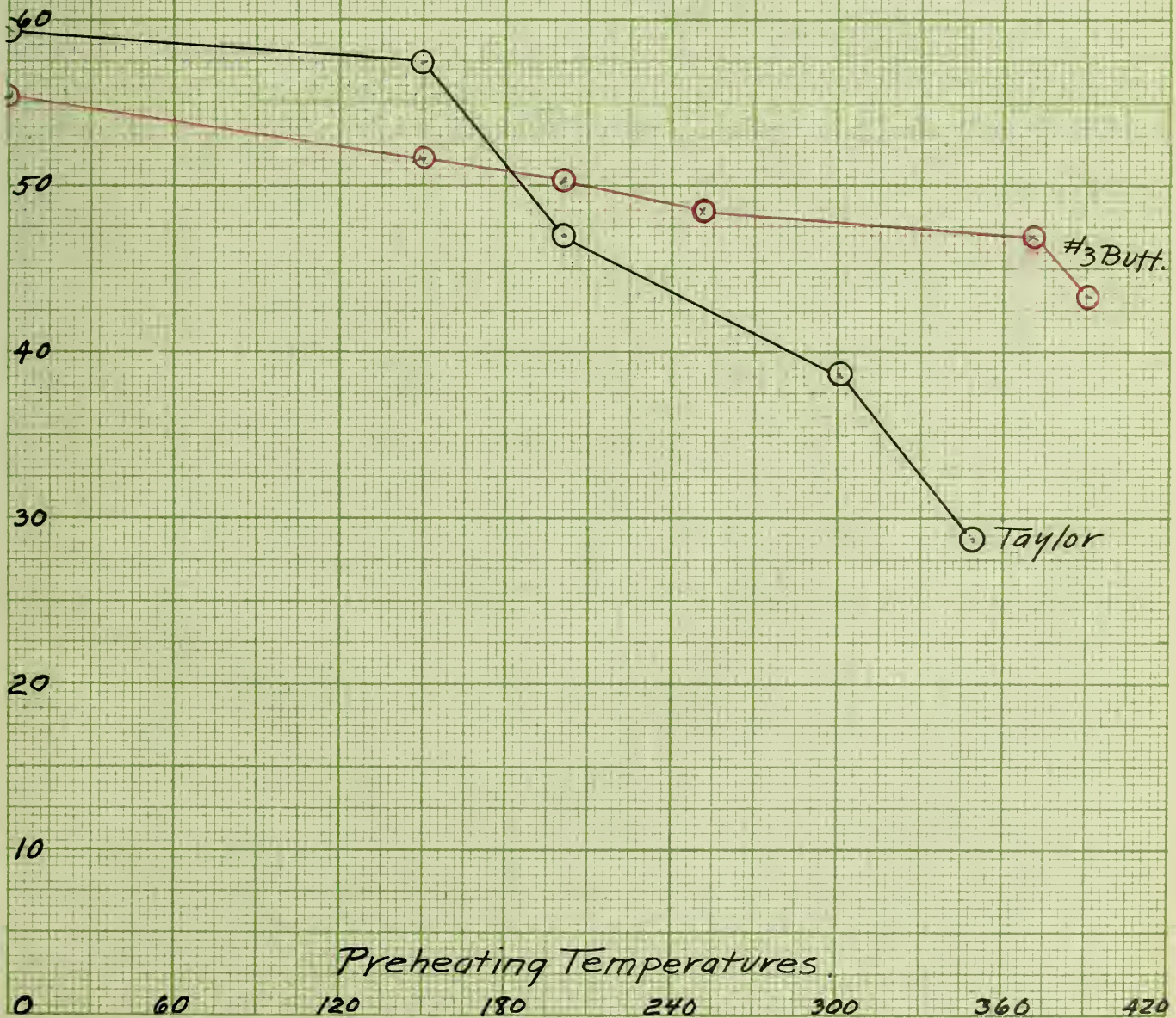




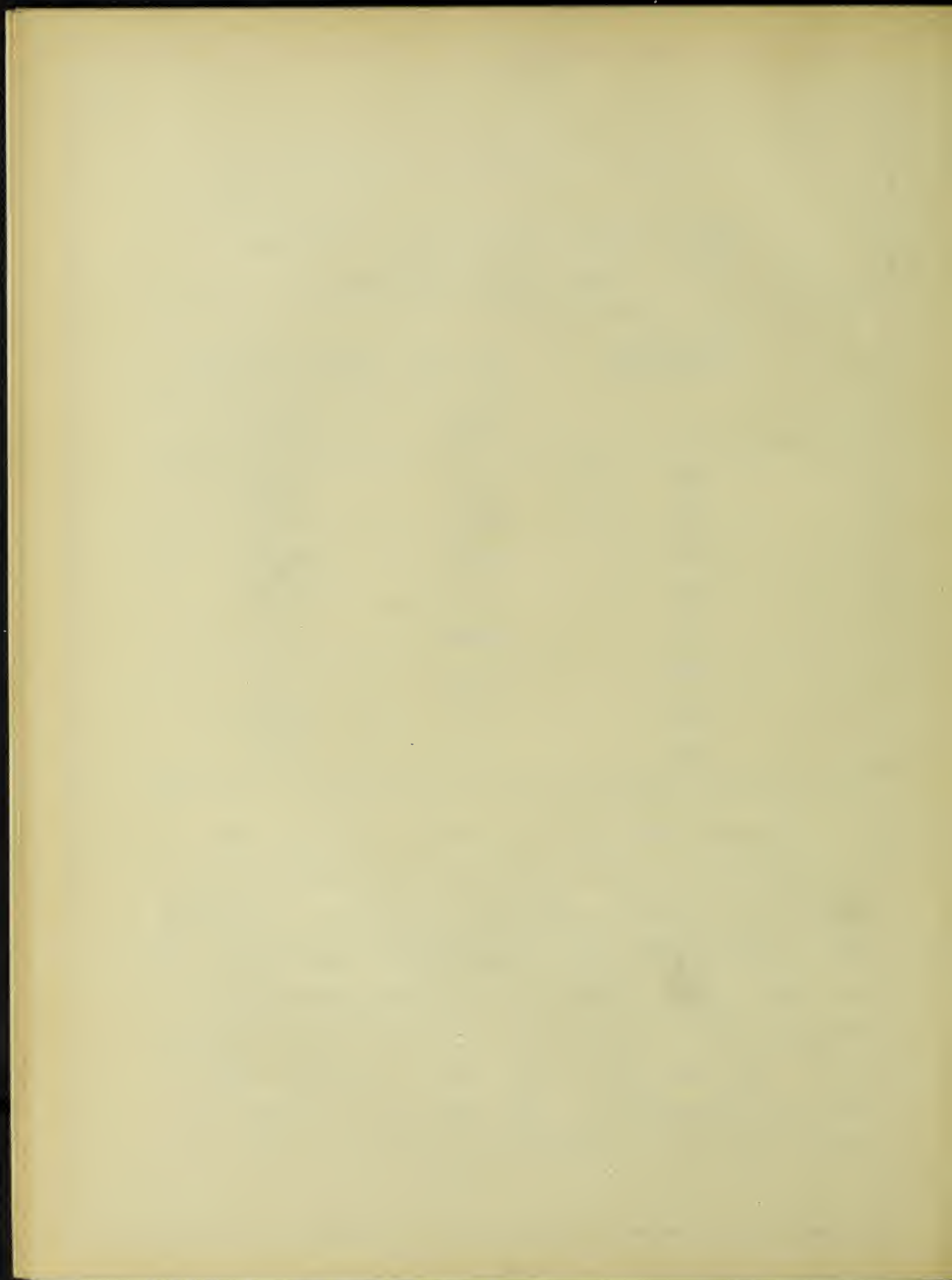


Table of Effect of Preheating on Shrinkage  
of Taylor and #3 Buff Clays.

Preheating Temperature	Per cent. Volume Shrinkage.	
	Taylor	#3 Buff
Raw	59.32	55.42
150	57.58	51.61
200	46.87	50.36
250	-----	48.46
300	38.64	-----
350	28.82	-----
370	-----	46.70
390	-----	43.35

Preheating had a much more decided effect upon the shrinkage of the clay than the addition of reagents and it affected the Taylor Clay more than the addition of reagents and it affected the Taylor Clay more than the #3 Buff. As was to be expected, increase in temperature decreased the shrinkage up to the point where the plasticity was entirely lost. The total effect in the case of the Taylor Clay was about 30% reduction. Upon wetting the preheated samples there was an evolution of heat which in some





cases was considerable altho no attempt was made to measure the amount of heat evolved. It was caused by the hydration of the dehydrated clay.

#### THE EFFECT OF ADDITION OF GROG UPON DRYING, CRACKING AND REDUCTION OF SHRINKAGE.

The grog used in these tests was made by calcining the clays to about 800-900 degrees C, the object being to eliminate all plasticity and oxidize them. Grog made from the Taylor clay was added to the Taylor plastic and grog made from the #3 Buff was added to the #3 Buff plastic, so that when the trials were made their behavior would not be influenced by the presence of foreign material. After calcining the grog it was put through a Braun Planetary Pulverizer, so that it all passed a ten mesh screen. Screen tests were made on both samples of grog to determine the fineness of grain. The results of the screen tests are below:





## Taylor Fire Clay

---

10-20 mesh	74.18%
20-40 "	14.40%
40-60 "	4.65%
60-80 "	1.00%
80-100 "	1.00%
100 up "	4.50%

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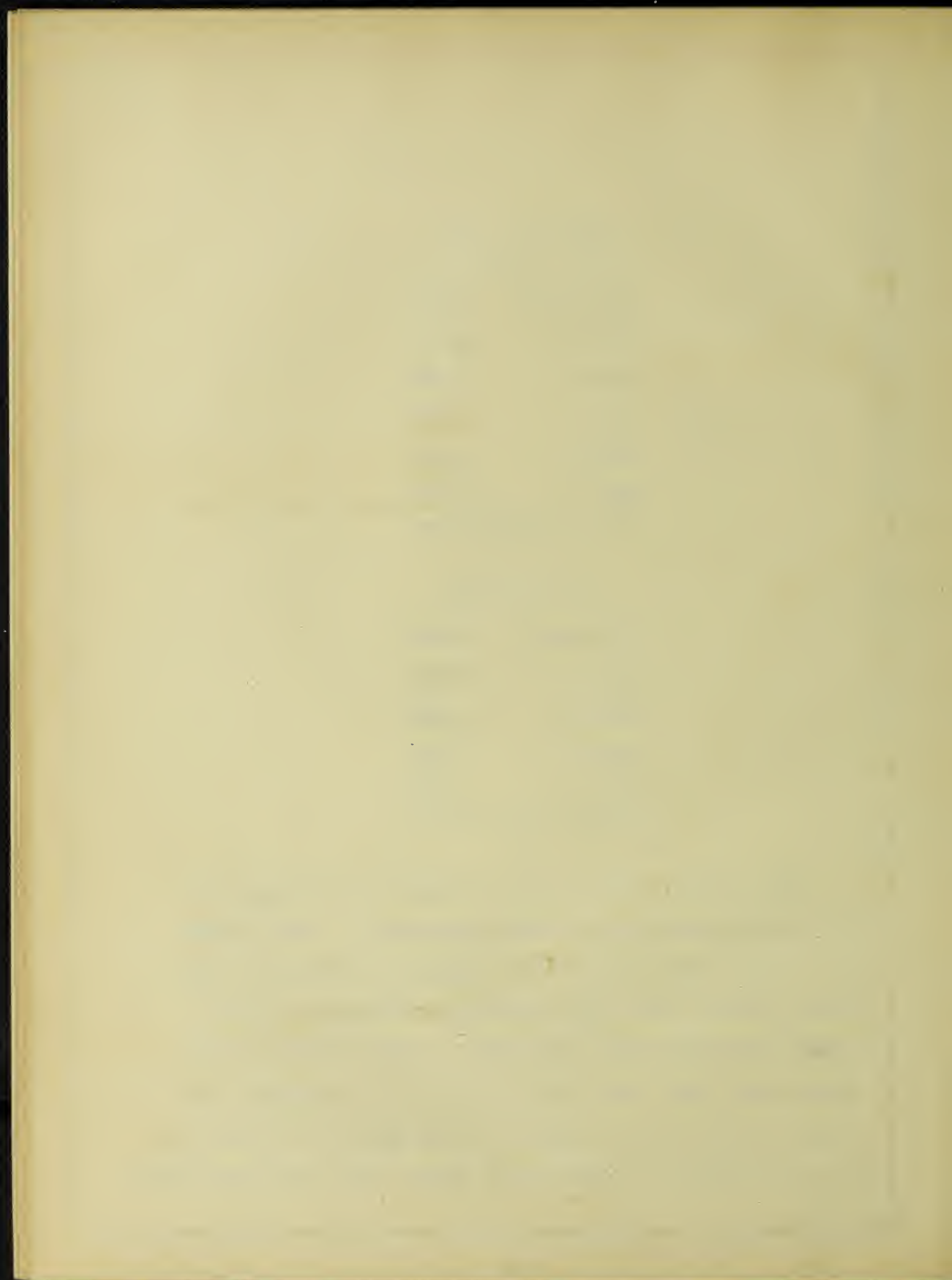
## #3 Buff Clay

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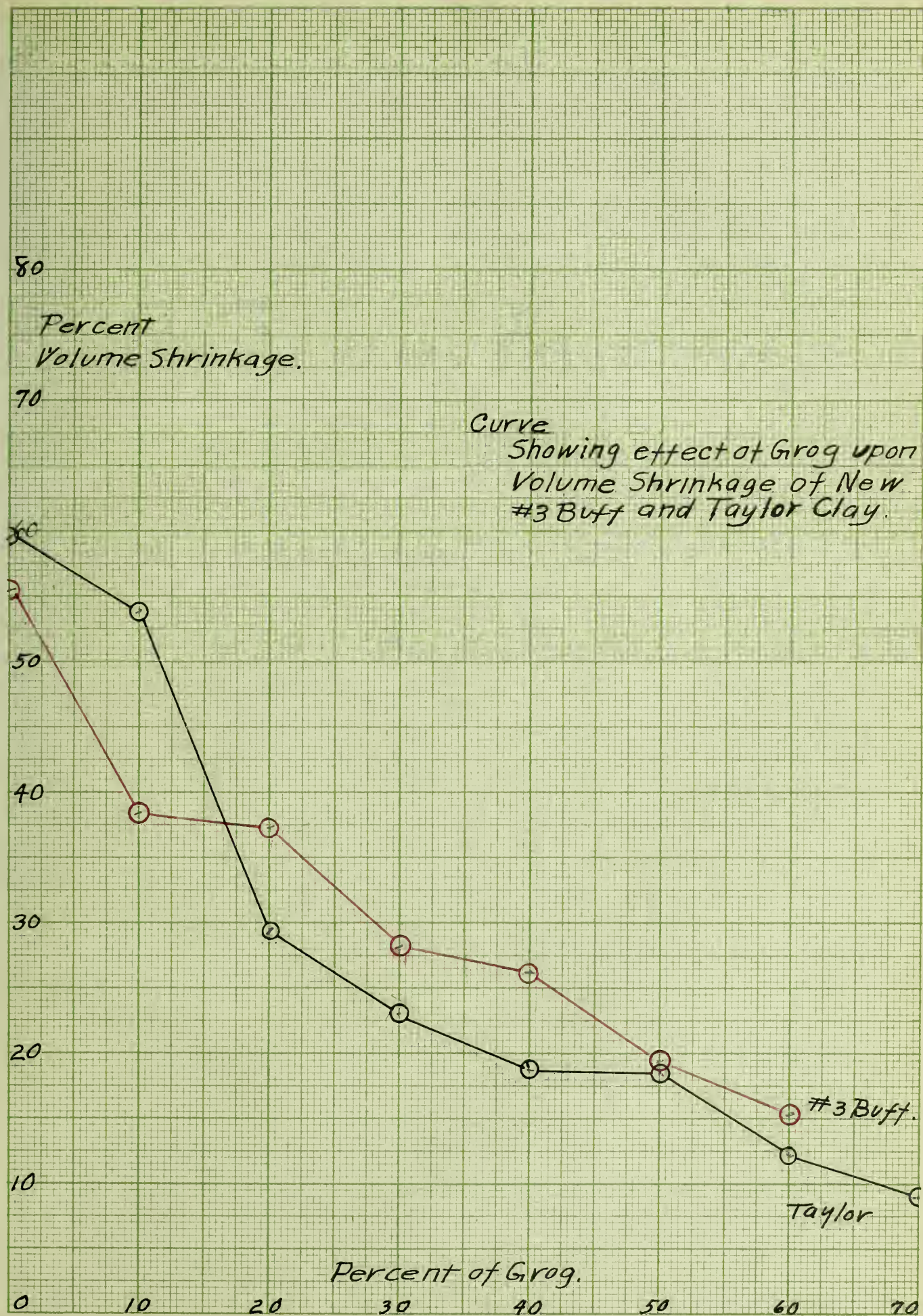
10-20 mesh	55.70%
20-40 "	24.70%
40-60 "	8.03%
60-80 "	1.40%
80 up "	9.07%

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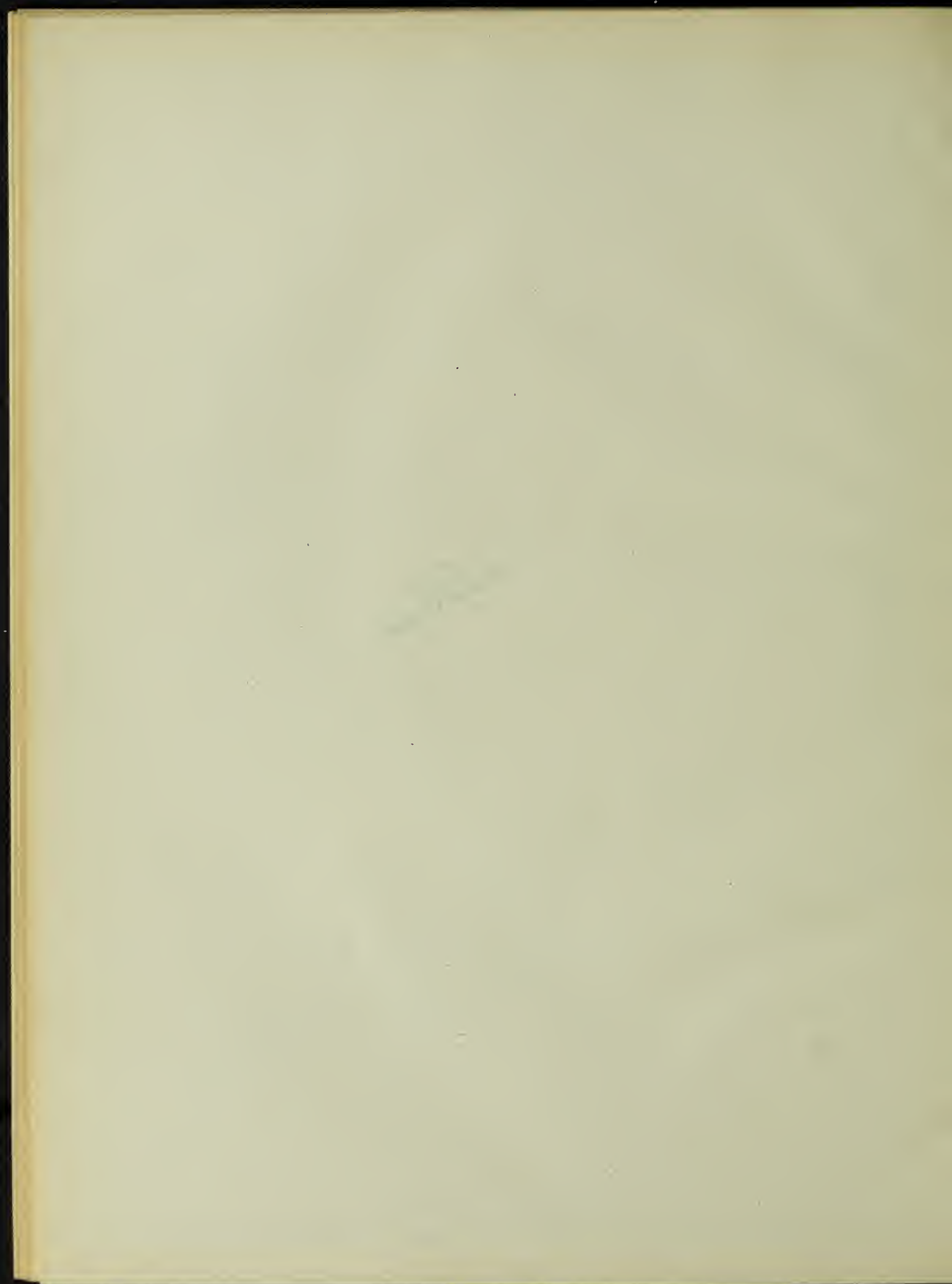
The most grog that could be added to the Taylor and #3 Buff Clays was 70% and 60% respectively. The amounts were varied from 10% up to the limits at intervals of 10%. These mixtures were made into small brickettes and their volume shrinkages were determined. One trial from each mixture was taken from the mold in the wet condition and placed directly in the drier at 110 degrees C., in order to test its ability to stand quick drying, this being considered







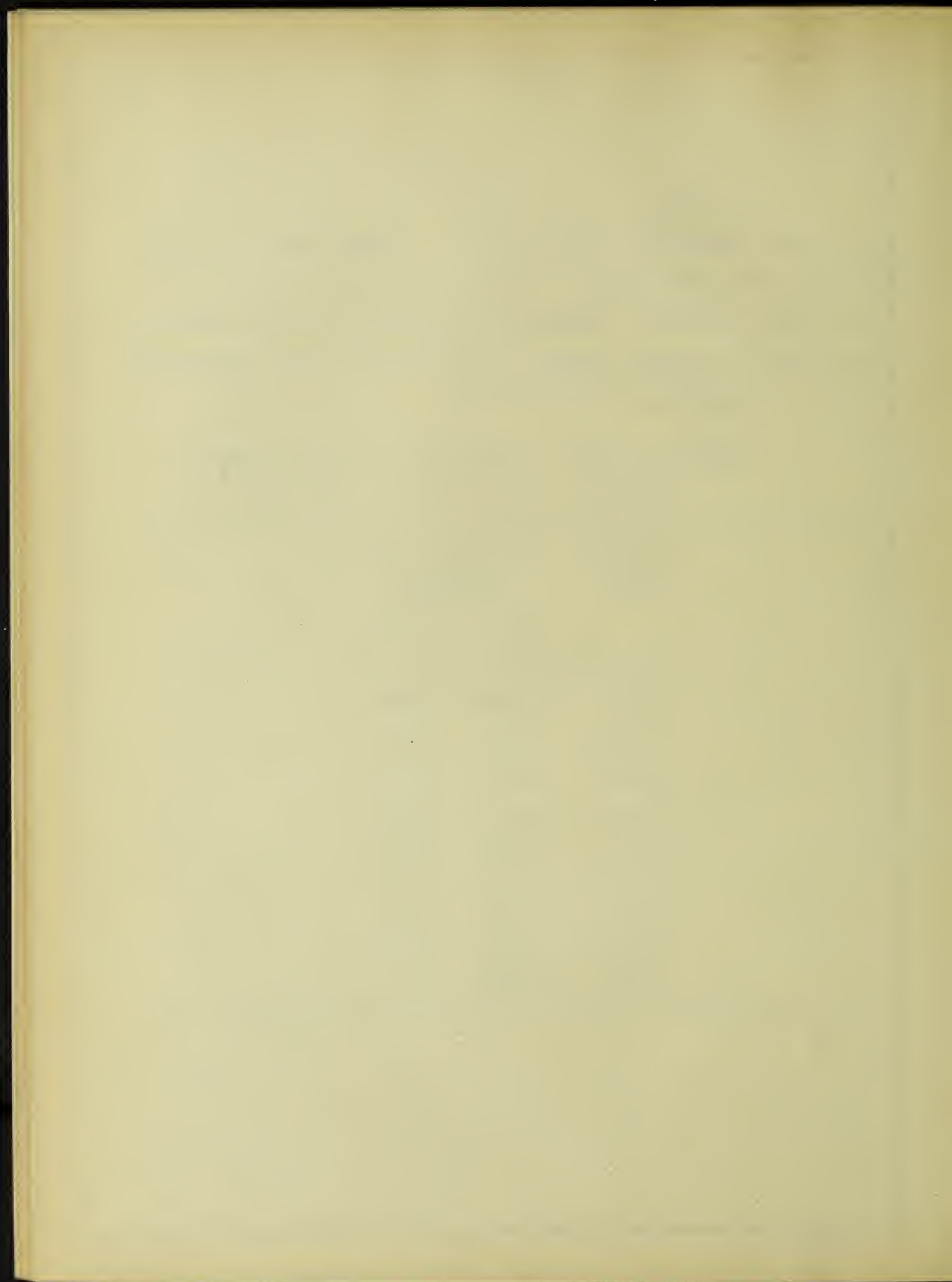




a rather severe test for the clays. Those pieces containing 60% or more grog, showed no warping or cracking whatever and dried quickly. As the grog decreased from 60%, cracking increased in direct ratio. The results are shown graphically by the accompanying curves and data:

Table of effect of addition of grog upon the shrinkage of Taylor and #3 Buff Clays.

Per cent. grog	Per cent. volume shrinkage.	
	Taylor	#3 Buff.
00.00	59.32	55.42
10.00	53.89	38.35
20.00	29.43	37.33
30.00	23.07	28.20
40.00	18.75	26.30
50.00	18.71	19.40
60.00	12.21	15.34
70.00	9.02	-----





### DISCUSSION OF THE THREE METHODS OF REDUCING SHRINKAGE.

Of the three methods used to reduce shrinkage and improve the drying behavior of the clays, viz: addition of reagents, preheating and addition of grog, the latter is the only one in which the effect was sufficient to recommend it to practice in working these two clays. By adding NaOH, NaCl and  $\text{CaCl}_2$  the shrinkage was affected slightly and in the case of the chlorides, perhaps a slight improvement was made in reducing the cracking during drying, but the benefits were so slight that they could not be considered of practical value.

By preheating, the shrinkage was reduced from 60% to 29%, but even 29% drying shrinkage is too much for safe manufacturing purposes and some further reducing agent or means had to be found. Further more the process of preheating presents many difficulties when put to a practical use. At about 400 degrees C. the clay suddenly loses all plasticity. Therefore it cannot be heated above this temperature. At the same time if the treating temperature is kept too low the full benefit of the process is not realized and the clay even after preheating will still have a high shrinkage. For these two reasons all the contents of the drier must be kept at about 350 degrees C. at all



times. In a large commercial drier it is quite difficult to maintain such conditions satisfactorily and cheaply.

It occurred to the writer that a much more practical way would be to calcine a part of the clay until devoid of all plasticity and until it was thoroughly oxidized, then add a definite amount of the raw clay to give it the required bonding power and working qualities. This of course would simply be the old process of adding grog.

By using the preheating process there is bound to be an uneven distribution of temperature and consequently an uneven intensity of preheating temperature, which would of course mean an uneven shrinkage. Furthermore, when the preheated clay is pugged with water it slowly regains its plasticity and high shrinkage. Consequently, unless it is molded soon after pugging, the effect of preheating is lost. On the other hand by using calcined clay or grog, definite and uniform mixtures could be produced at all times. This led to a series of experiments upon the action of grog on this particular clay, which proved to be (as the results will show), a very effective practical means of reducing the shrinkage. By adding 70% grog to the raylor clay the shrinkage was reduced from 59.32% to 9.02% and the brickettes could be dried quickly at 110 degrees C. without cracking or warping. Using 60% grog, full sized soft mud bricks were made, dried





and burned at cone 12 successfully without cracking, warping, or checking. This appears to be the only effective practical means of using this clay in the manufacture of clay products without disastrous drying and burning losses.

#### INVESTIGATIONS IN REFRACTORY PROPERTIES.

There are several different tests that are used to determine the refractory properties of clays; each one of which is a good index to those properties, but if used alone is not extensive enough to give full information.

The chemical analysis is often applied and while it shows the amounts of the constituents and will tell in a general way whether a clay can be classed as a refractory or non-refractory; it does not determine physical properties, such as fineness of grain, which enter into the consideration and so materially affect the refractory behavior. Oftentimes clays of nearly the same chemical composition will behave quite differently as regards refractoriness and oftentimes the fusion points of evidently inferior clays will be quite high.<sup>1</sup>

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1. Ill. State Geol. Survey. Bull. No. 4 P. 133.





The fusion test in which a sample of the clay is subjected to the influence of heat will bring out properties that the chemical analysis never would indicate. In this test small pyramids of the clay to be tested are placed close up to Seger Cones of known composition and known fusion temperatures and the refractory quality of the unknown clay is determined by comparison of the effect of the heat on it with the effect of the heat on the known cones. However, these cones are very small and very light and for this reason the viscosity of the test cone will hold it up longer than another cone of perhaps equal refractoriness but less viscosity or longer than the cone material would stand up if a load were imposed upon it, as is the case in actual practice. "It is evident, therefore, that the determination of the so-called melting point of a clay or fire brick though useful in differentiating between low and high grade materials, does not offer a reliable means of expressing the entire refractory behavior." 1

In order to supply a test where the clay would be under a given load at a given temperature, a special load test kiln was built at the U. S. Bureau of Standards at Pittsburgh, Pennsylvania. Loads and temperatures that most nearly approximate conditions in practice were chosen and the bricks were put thru the tests. Two sets of conditions were tried out. They were that a nine inch fire brick must not be

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1. Trans. Amer. Cer. Soc. Vol XII.

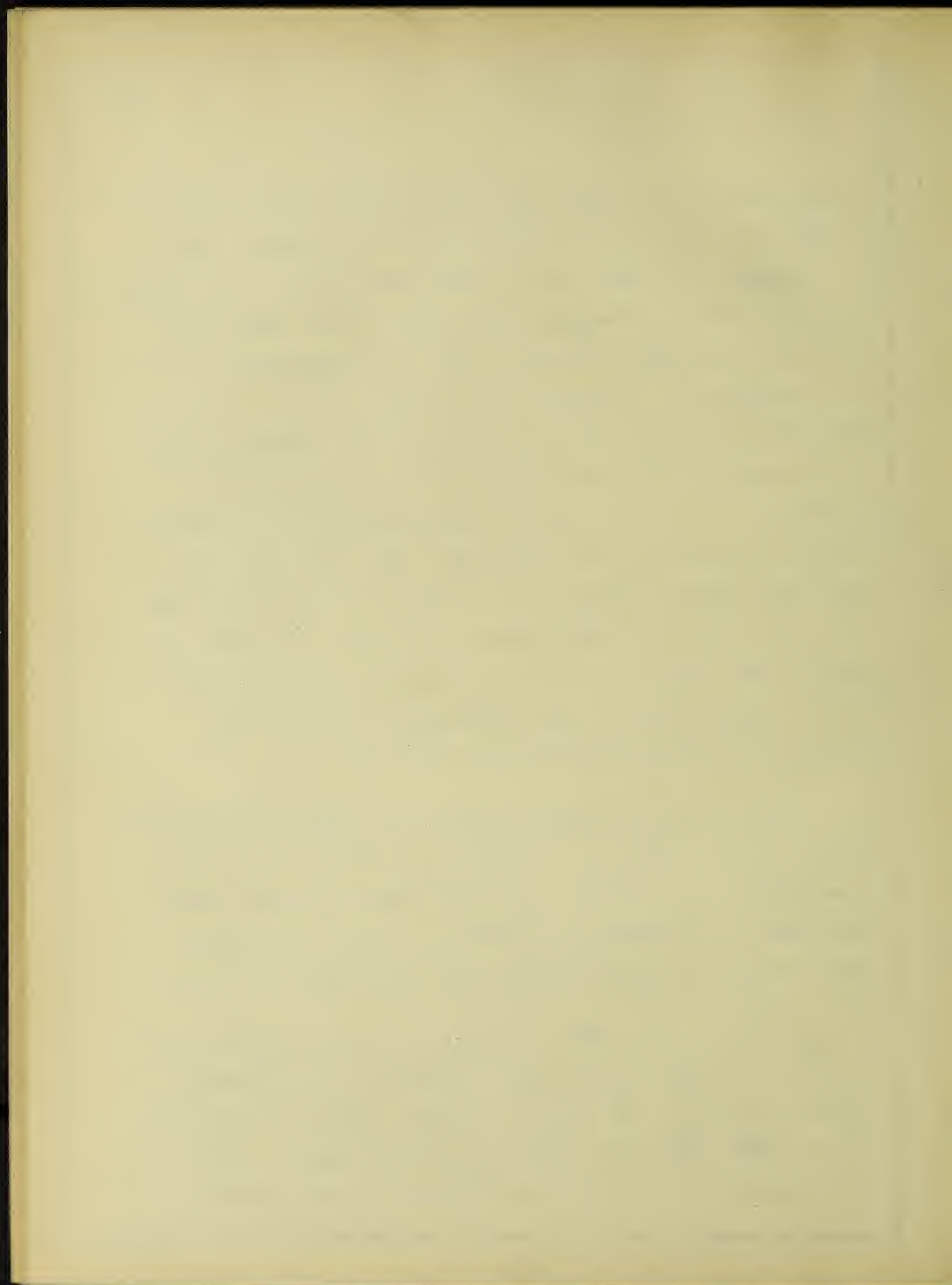


compressed more than one inch, either under a load of 75 pounds per square inch at 1300 degrees C. or under a load of 50 pounds per square inch at 1350 degrees C. The standard government test now requires that a No. 1 fire brick carry a load of 50 pounds per square inch at a temperature of 1350 degrees C. In practice the average burden is about 25 pounds per square inch, but owing to the influence of the time factor in the latter case the load is increased to balance it. The work was done by Mr. Bleininger and altho he did not claim that any brick that would not stand the test was totally worthless as a refractory he did claim that any brick that did stand it was a first class brick for use under load conditions and that the amount and fusibility of the least refractory clay constituent governed the behavior of the brick under load conditions.

Another test of refractoriness is the Porosity-Temperature curve which will show the progress of vitrification as the temperature is increased. It is of course essential that a good fire clay maintains its porosity at high temperatures and the higher its vitrification point the better the clay.

If all of these tests are applied to a fire clay it is possible to form a comprehensive opinion of the refractory properties of the clay from all angles. As far as possible all of these tests were carried out in this clay. Owing to the fact that our load test kiln was not yet completed,





that test could not be made, and it was not possible to burn trials as high as was desired for the Porosity-Temperature determination.

### CHEMICAL ANALYSIS

Taylor Fire Clay.

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Constituent	Per Cent.
SiO <sub>2</sub>	52.40
Al <sub>2</sub> O <sub>3</sub>	19.08
Fe <sub>2</sub> O <sub>3</sub>	9.07
CaO	1.80
K <sub>2</sub> O & Na <sub>2</sub> O	1.29
MgO	trace
Mech Water	5.85

---

Total RO fluxes = 13.07%

A complete analysis of the #3 Buff clay was not made although some of the constituents were determined, as follows:

SiO <sub>2</sub>	58.96%
Al <sub>2</sub> O <sub>3</sub> :	
& : :	
Fe <sub>2</sub> O <sub>3</sub> :	29.21%
CaO	1.83%



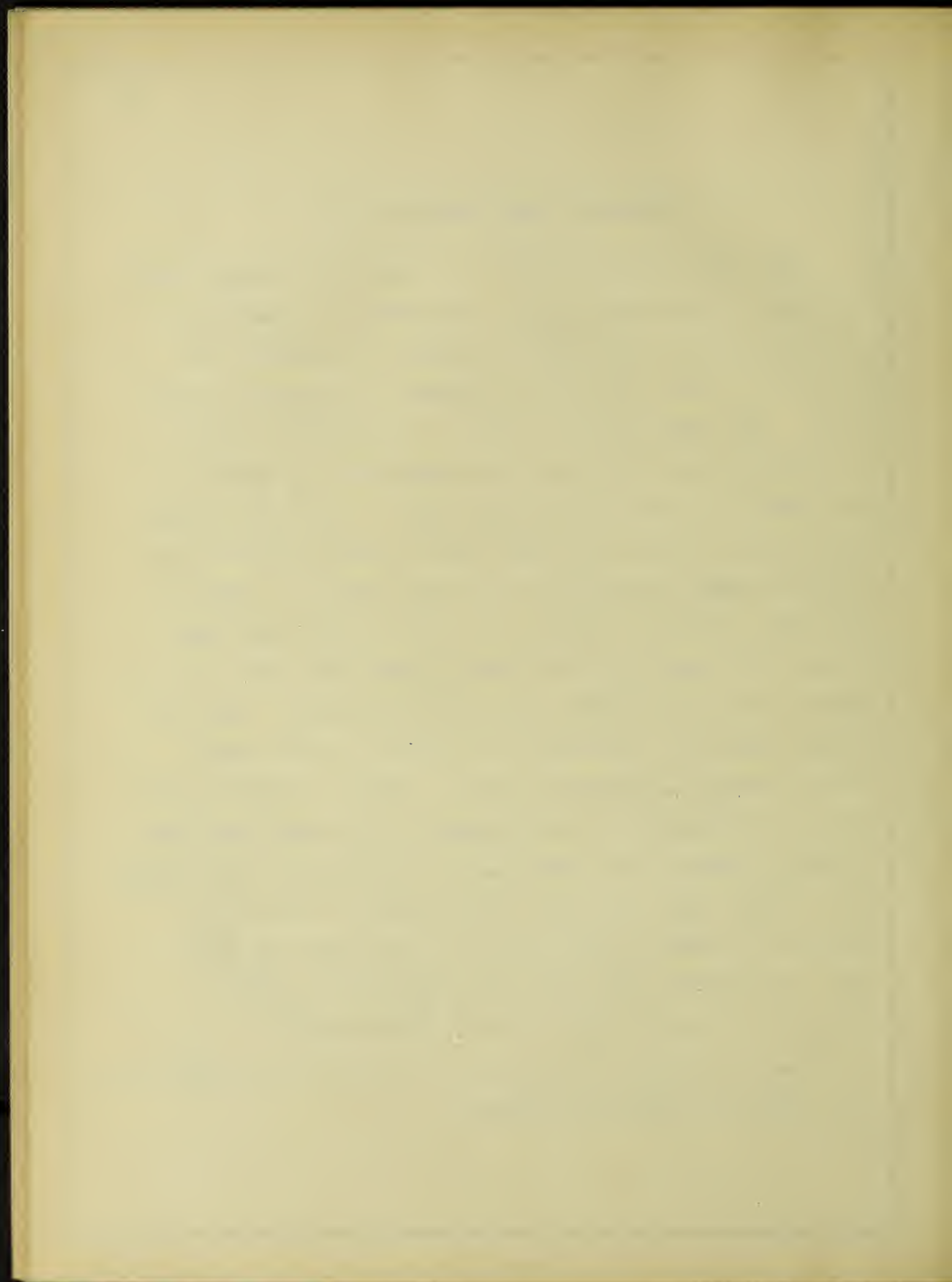


## SOFTENING POINT DETERMINATIONS.

The fusion tests were made in a small pot furnace heated by means of a Fletcher burner. Very small cone pats were employed and the cones placed as close to one another as possible so that they would all receive the same heat treatment. The pats held three cones. In the middle was the cone whose fusion point was to be determined and close on each side of it was a known Seger cone. The fusion points were found to be for the Taylor Clay at cone 18 and for the #3 Buff between cones 18 and 19 or about 18-1/2. Owing to the high content of iron the clay cones bloated very badly at the high temperatures and several check tests had to be made in order to determine the points accurately. The two fusion points are practically the same and are far below those expected in a good fire clay. In fact, comparing them with Mr. Bleininger's classification of fire clays according to fusion temperatures, they fall far short of his requirements for a number Three fire clay, which specify a fusion temperature of between cones 25 and 30, and he does not even call these Number Three Fire clays refractories altho he says they are used to some extent in boiler work.<sup>1</sup>

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1. Notes from classroom lectures.



## LOAD TEST.

It would be useless to put this brick to the load test. 1350 degrees C. is equivalent to cone 11 and only seven cones below the temperature at which the clay fuses as a cone. In his work on "The Behavior of Fire Bricks under Load Conditions, Mr. Bleinenger says, "A brick softening below cone 30 would not stand much of a chance in a load test."<sup>2</sup>

## POROSITY - TEMPERATURE CURVE.

Porosities were determined by using the wet, dry and suspended weight process, in the following formula:

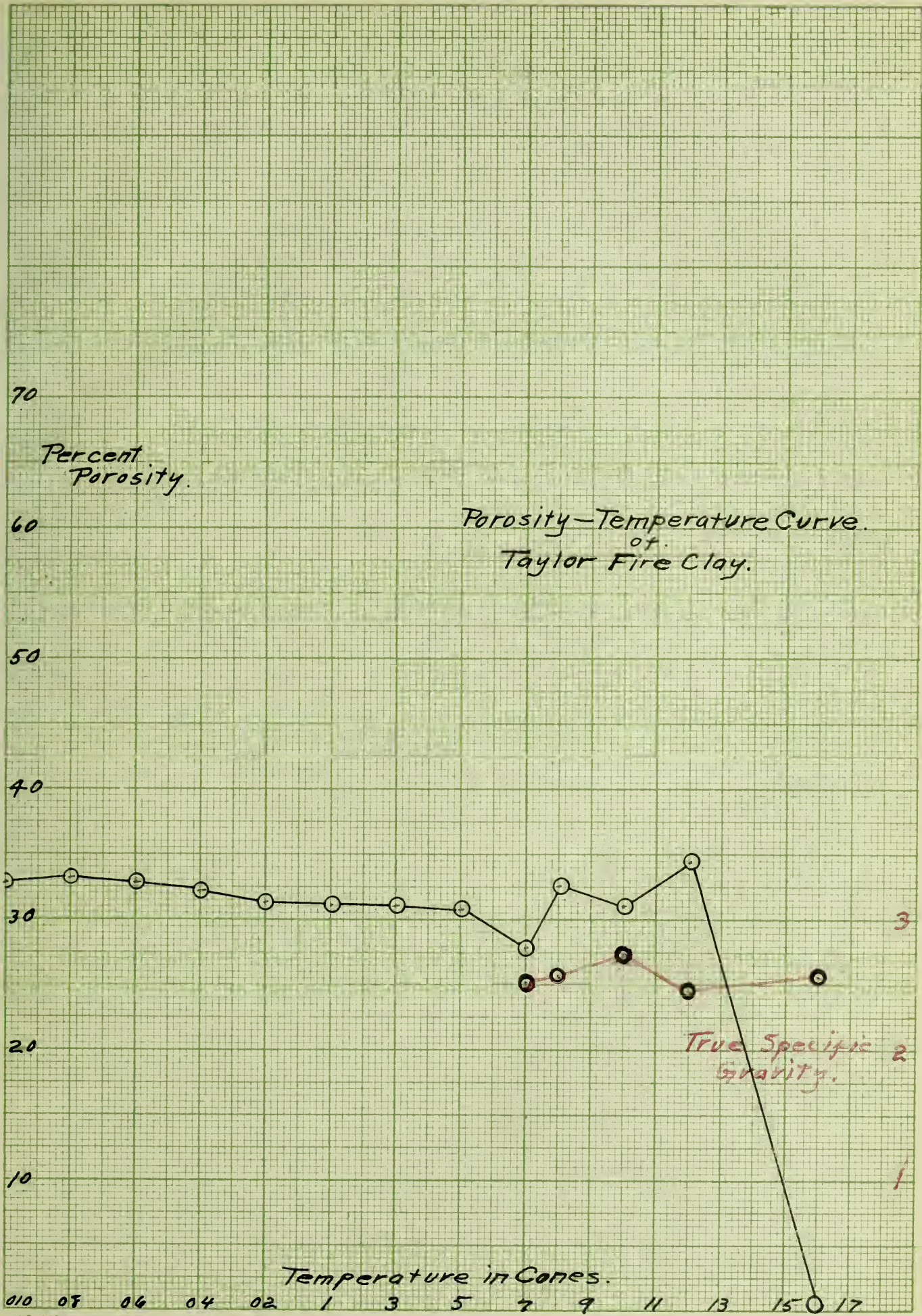
$$\frac{\text{Wet Weight} - \text{Dry Weight}}{\text{Wet Weight} - \text{Suspended Weight}} \times 100 = \text{Per cent. Porosity.}$$

The clay containing 60% of its own grog, was made into the same kind of brickettes as those used for determining volume shrinkages. The trial pieces from cone 010 to cone 3 were burned in a draw-drial burn in the large test kiln. Those from cone 5 to cone 12 were placed in the large test kiln with other burns that were going to their required temperatures. The cone 16 trials were placed in a small, round, fire-clay sagger and burned in a Fritt pot with a Fletcher burner. Four cones were put in the pot all around the trials and cone 16 was registered in all parts. These trials were placed on end in the pot and when taken out they were stuck to the bottom and leaning over in a badly fused and bloated condition.











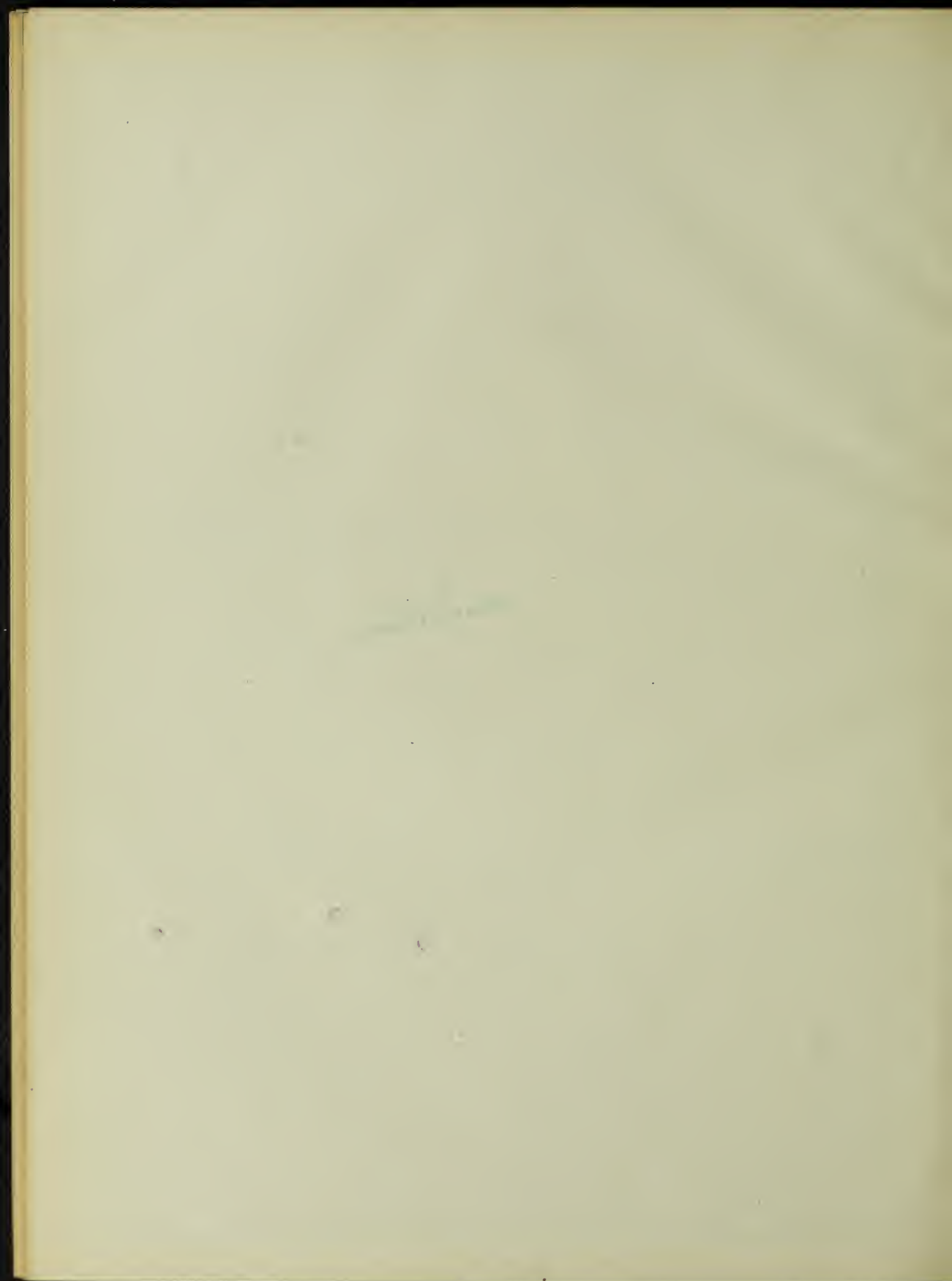




TABLE OF POROSITY AND TRUE SPECIFIC GRAVITY.

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Temperature in cones	Per cent Porosity	True Specific gravity.
<hr/>		
010	32.78	-----
08	32.21	-----
06	32.93	-----
04	32.21	-----
02	31.39	-----
1	31.09	-----
3	31.06	-----
5	30.86	-----
7	27.85	2.507
8	32.67	2.567
10	31.00	2.745
12	34.44	2.440
16	00.598	2.565

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The points located above cone 5 on the Porosity - Temperature curve do not follow a regular path. However, this was probably due to a little irregularity in the heat treatments, within the range of vitrefication, where slight differences make such a marked change in a clay, subject to such sudden fusing action as this clay shows.

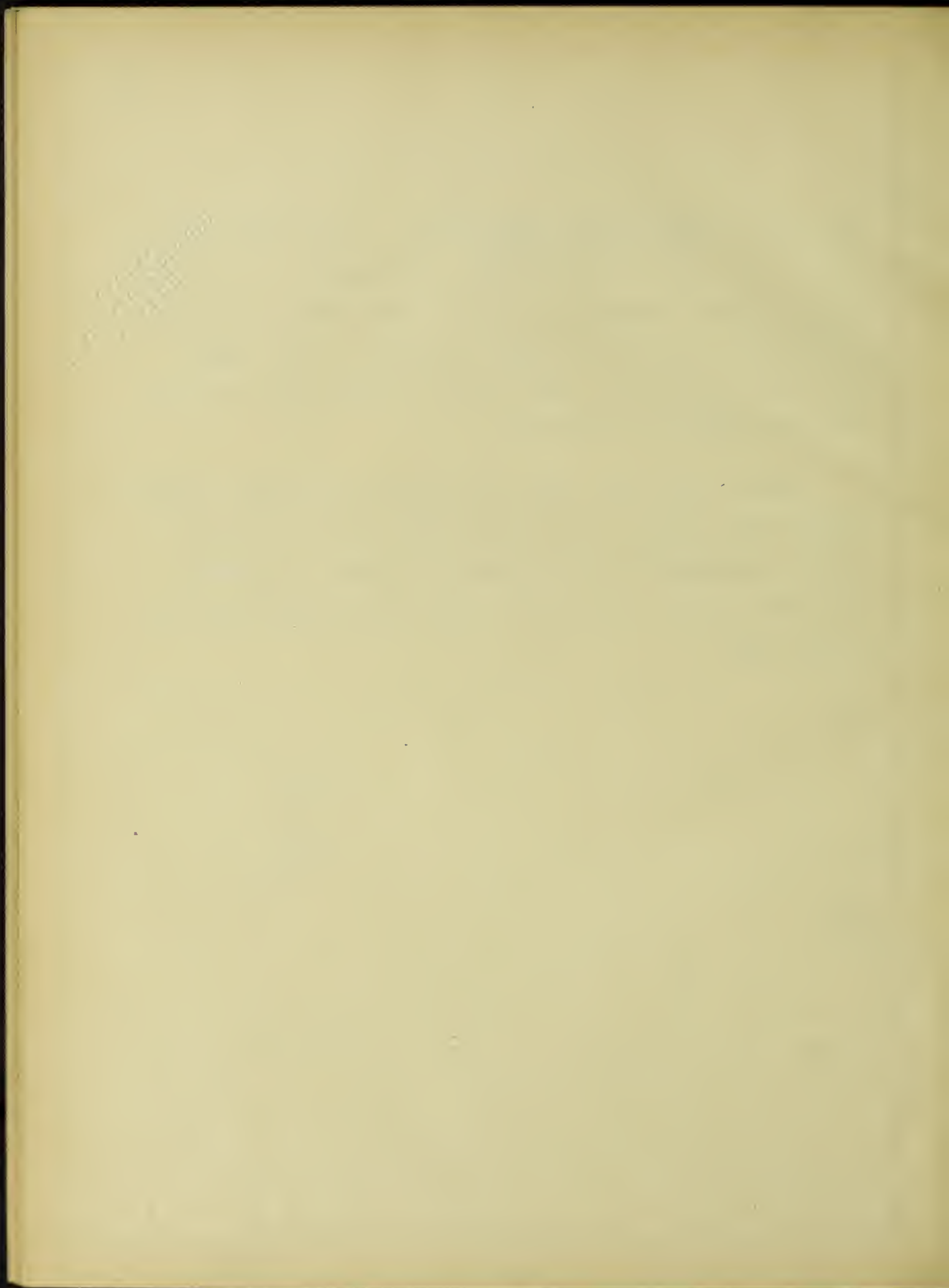
In order to check up these differences the true specific gravities were determined by means of the Pycnometer and the



per cent of bleb structure calculated. It was found that at cone 7 the trials contained 9.28% bleb structure, at cone 10 they contained 11.41% and at cone 16, they contained 31.85% so that while this trial showed a porosity of only .598% its true porosity was 31.85% owing to bloats into which the water was unable to penetrate.

These curves show that a very sudden and violent fluxing action takes place in the clay at about the temperature to which a refractory is most commonly subjected in practice, and emphasize more forcibly the very poor refractory quality of the clay.



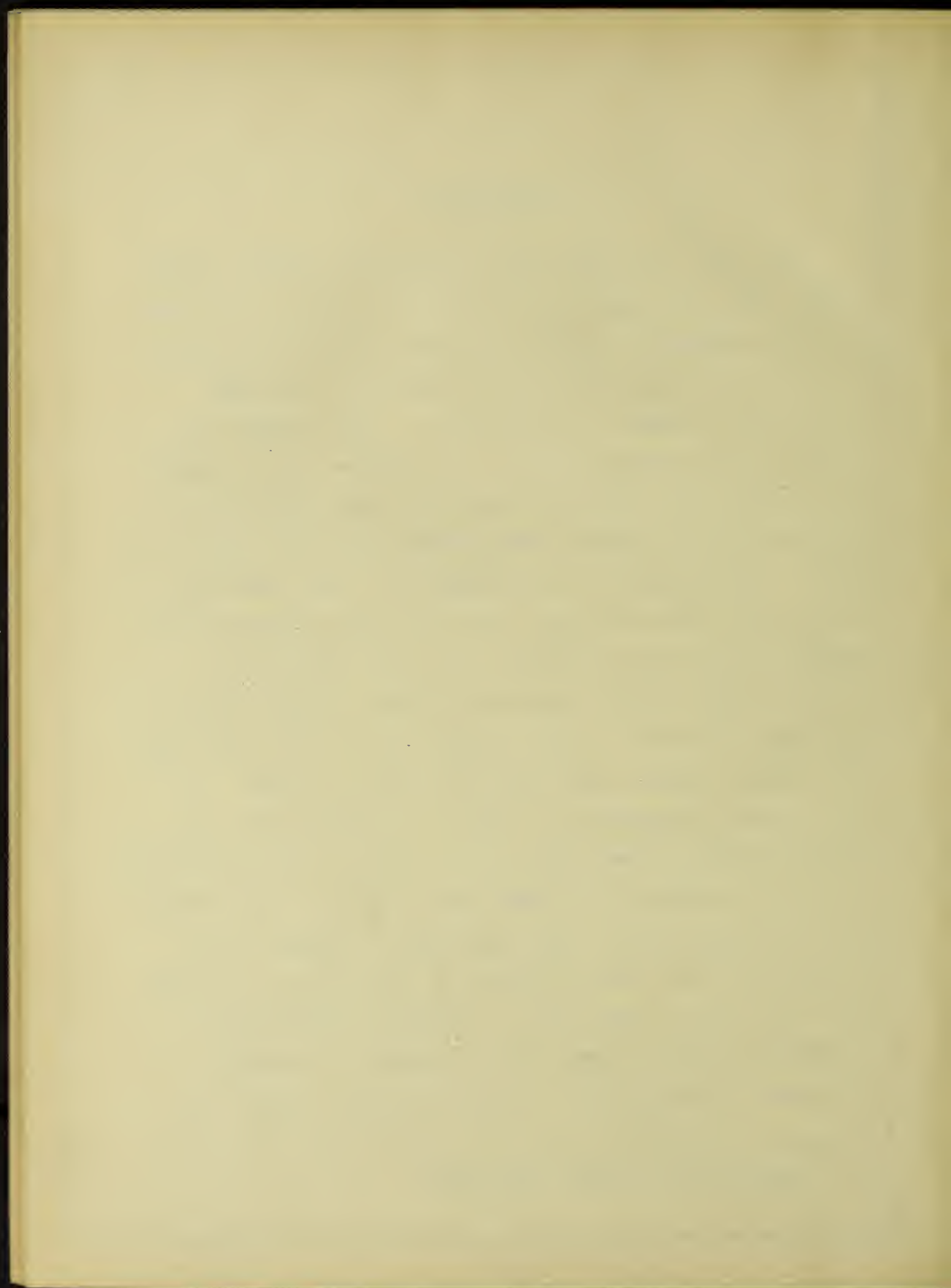


## CONCLUSIONS.

The ideal fire clay would be one of the formula,  $\text{Al}_2\text{O}_3$ ,  $2\text{SiO}_2$ ,  $2\text{H}_2\text{O}$ , with sufficient plasticity to insure good working qualities and at the same time have a fusion temperature of cone 32 or 33 and stand up well under the load test. This is the formula of a pure kaolin. Clays having all five of these characteristics; viz; freedom from RO fluxes, high fusion point, ability to carry the load, good working qualities and the proper silica-alumina ratio are very rare. The presence of the RO fluxes in a clay, lower its fusion temperature decidedly and at the high temperatures to which the clays are subjected, a very small quantity of flux will have a most deleterious effect. A clay containing high silica and low flux is much better than a clay with the correct silica-alumina ratio but high in fluxes. As the silica is increased in a clay of the kaolin type, the refractoriness is decreased, but the silica is not as effective in this direction as the fluxes and the effect of the silica is often exaggerated. "In a clay high in fluxes, the refractory qualities can be improved by the addition of silica, but high silica - high flux is a dangerous combination;"<sup>1</sup> The state and size of grain of the fluxes in a clay are important considerations. They are much more active in a

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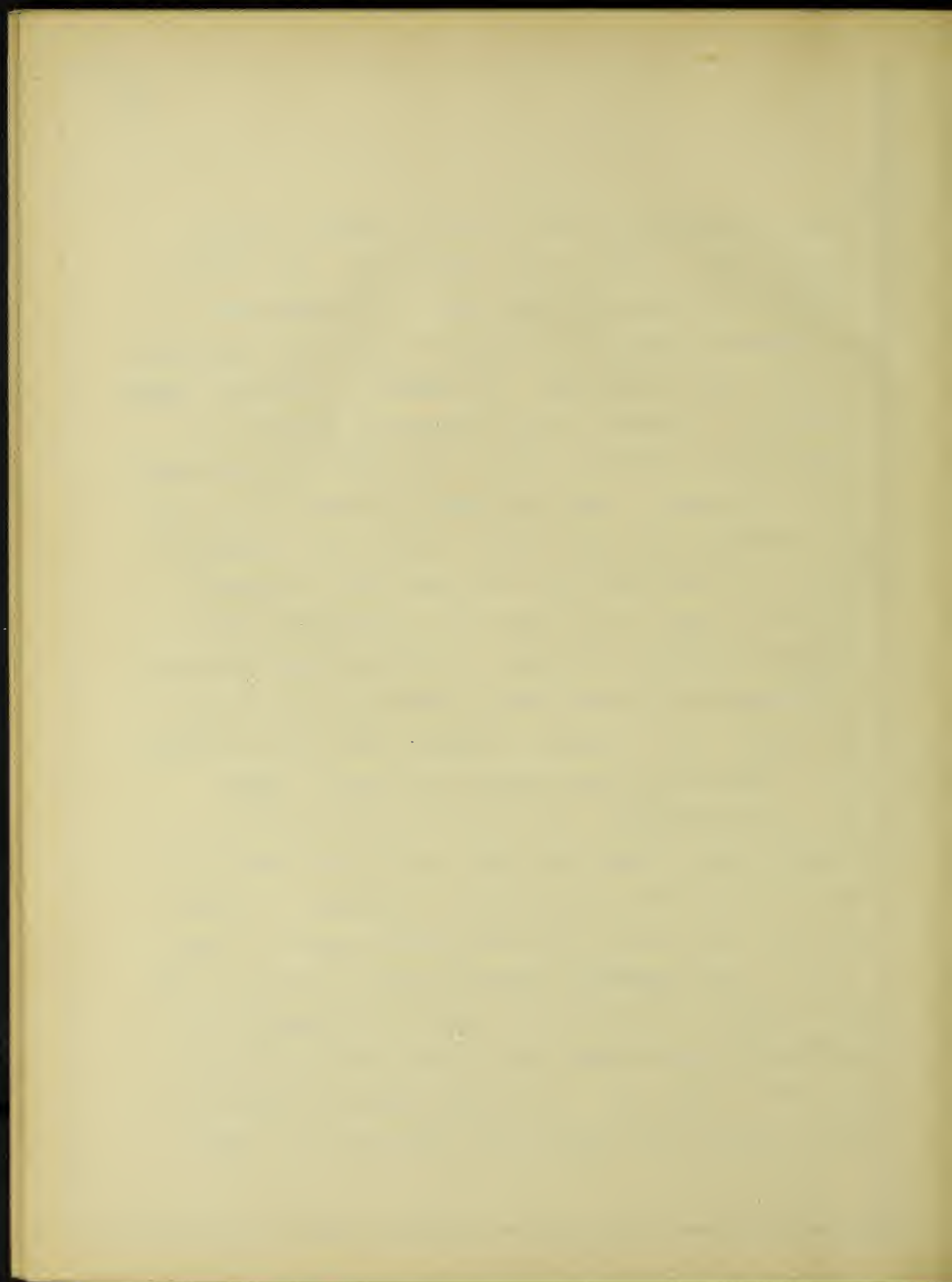
1. Trans. Amer. Cer. Soc. Vol XII. P.





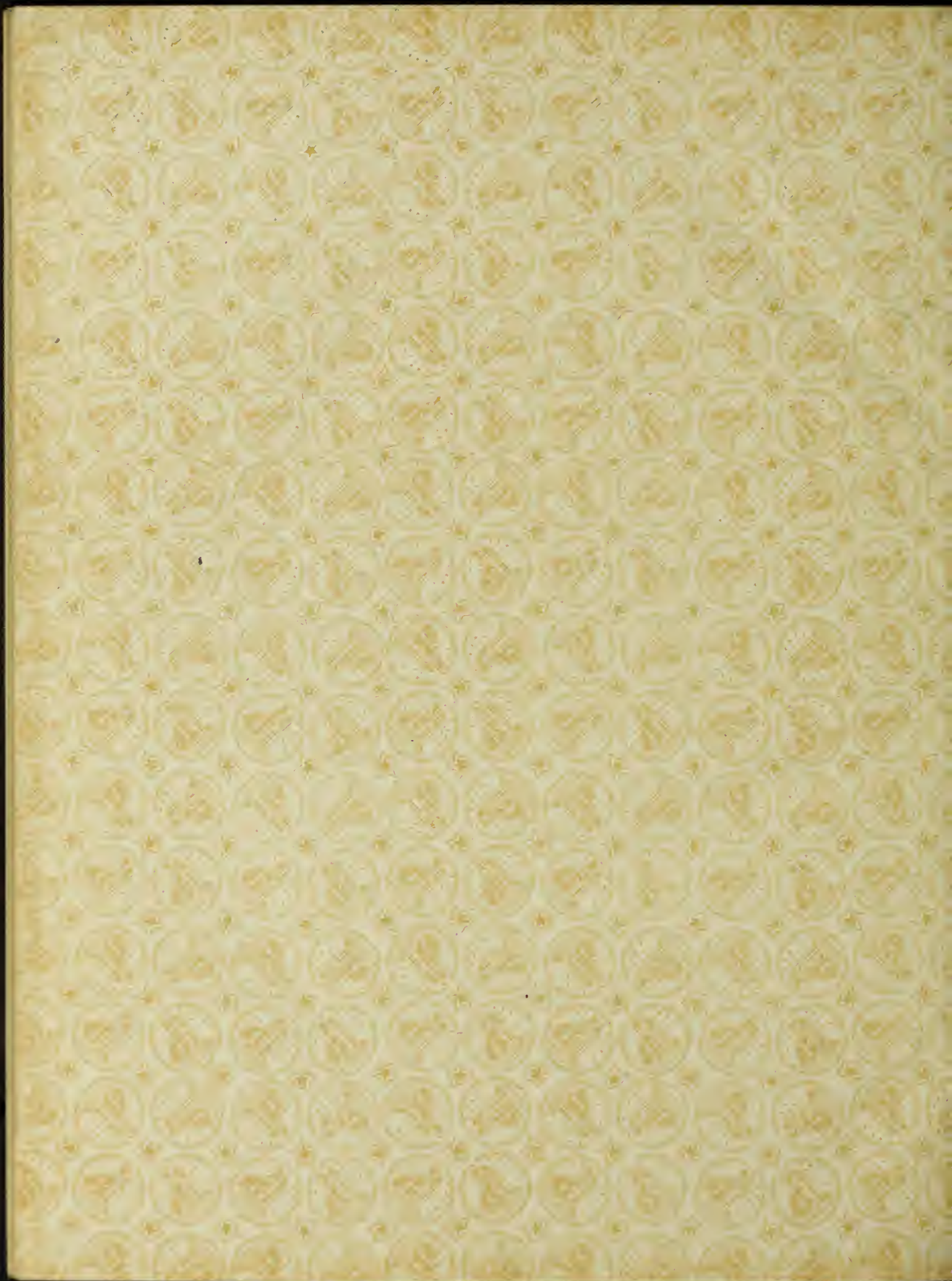
finely divided state, while if iron is present in coarse granular form, it will do very little harm. The maximum allowable RO content including iron is 0.22 equivalent.

The molecular formula of the Taylor clay has this combination of high silica - high flux. The formula is, .6723 RO,  $\text{Al}_2\text{O}_3$ , 4.67  $\text{SiO}_2$ . A glance at this formula would lead one to predict that it would not stand a very high fusion temperature, which is the case. This clay would be of very little value as a refractory when used alone. However, the refractory behavior of bricks made from this clay might be somewhat improved by using it as a bond clay in conjunction with a high grade calcined flint clay. All flint clays shrink much in drying and burning and owing to their low bonding power they crack. This excessive shrinkage is cut down by calcining. Calcined flint clay makes a far superior product as it is much stronger. It is very important to get an intimate mixture between the flint and the bond clays, so that the flint particles will have an opportunity to unite with each other and hold when the bond clay begins to fuse. If they are not intimately mixed the bond clay fuses at comparatively low temperatures and acts as a lubricant to the unattached flint particles, thus causing the brick to fail soon under a load test. Fire bricks should be burned as high as possible and certainly not below cone 12. Brick



burned to high temperatures withstand load conditions in a wall much better than the softer burned bricks. There is also less shrinkage and cracks in the wall are avoided to considerable extent.



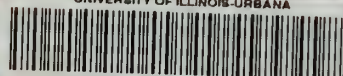








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